# TOXICITY OF THE PYROLYSIS PRODUCTS OF SPACECRAFT MATERIALS

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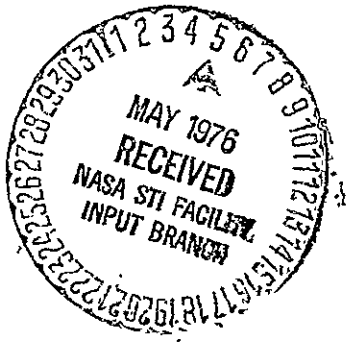


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## I. Introduction

The toxicity of pyrolysis or thermodegradation products of various types of materials is of considerable importance in making informed decisions on the selection of materials for specific uses. The hazards of such toxic fumes may be present under a number of circumstances, such as fires in warehouses, manufacturing plants, homes, offices or apartment buildings. It is now being recognized that some of the more dangerous (hazardous) gases generated by fires result from pyrolysis or thermodegradation of various polymeric materials. It must also be recognized that an open flame is not a prerequisite to generation of toxic gases from these polymers; electrical or other types of overheating may result in generation of toxic degradation products and their release to the atmospheric environment. While such toxicants may be hazardous to firemen in their attempt to control a building fire and to occupants of the building, these toxic vapors may pose even greater hazards to persons in a closed environment (e.g., spacecraft or submarines) or a semi-closed environment (e.g., airplanes).

It is not necessary to have an open fire to generate toxic fumes from a material; this may occur from a smoldering material or from electrical overheating such as a short circuit. Flammability of many materials may be markedly reduced by inclusion of various flame retardants. While this may help control one problem, that of open flame and heat generation, if these flame

retarded-materials are exposed to a source of sufficient heat they will still decompose and the fumes produced generally are more toxic than the comparable material without the flame retardant.

Thus, alleviation or solution of one problem may, in turn, create or intensify another problem. Which of the two problems is the most critical, or creates the greatest hazard, is largely dependent upon the use of the material (i.e., function, location and quantity of material used, the probability of it being exposed to a source of intense heat, where fumes would go if this should happen, etc.). Since there is generally a choice of two or more materials which will provide the desired physical characteristics necessary for the application, other factors (in addition to cost) should be considered before making a final selection. Among these 'other factors' should be potential for exposure to heat, relative toxicity of fumes from the candidate materials, if fumes are formed where would the fumes go (into an inhabited area or exhausted to the outside, etc.), the relative importance of non-flammability, etc.

Data from the work reported herein provides some of the information needed to make a rational, informed judgement in such materials selected. These data provide guides to (1) approximate temperature necessary to initiate thermodegradation of the materials tested, (2) the relative toxicity of thermodegradation products from the various materials, (3) the relative importance of carbon monoxide as the primary cause of death (as contrasted to cyanide or other toxic gases), and (4) whether or not the

hazards of the fumes are confined to the time of exposure, or whether post-exposure death is likely. Of the two experimental procedures employed, the NASA Procedure mimics a closed environment situation (without filters or adsorbents), while the MSTL Procedure more closely approximates a semi-closed environment situation, in which some fresh air is being continuously admitted.

## II. Materials & Methods

This past year there were nine material samples sent by NASA for evaluation of relative toxicity to rats of thermodegradation products from the samples. All nine of these were tested by the MSTL Procedure and three were also tested by the NASA Procedure. A list of these materials, along with their code designations, is presented in Table 1.

Methods employed in this study (MSTL Procedure and NASA Procedure of thermodegradation and rat exposure, carboxyhemoglobin determination, etc.) are the same as described in last year's Annual Report "Toxicity of the Pyrolysis Products of Spacecraft Materials", dated November 25, 1974, and will not be repeated here.

## III. Results

Upon receipt of a sample from NASA for evaluation, the first step is to determine its thermal profile by TGA (Thermogravimetric Analysis). This information is used in establishing thermodegradation temperature for the MSTL Procedure, and in some instances, modifies the maximum degradation temperature for the NASA Procedure. The TGA profiles for these samples under various experimental conditions are

presented in Figures 1 through 28, and analysis of these data is presented in Table 2.

A summary of the $LD_{50}$ values, based upon (1) deaths before removal of rats from the exposure chamber, (2) deaths occurring within 48 hours of exposure to fumes, and (3) deaths occurring within 14 days of exposure to fumes, by both the MSTL and NASA Procedures, where appropriate, is contained in Table 3. The mean and range of COHb levels of rats dying in the MSTL chamber is presented in Table 4, along with the CO concentration (determined by G.C.) as a function of temperature during pyrolysis produced by the $LD_{50}$ sample weight. Figures 29-31 show the relationship between COHb levels and % mortality for these samples. During or following pyrolysis of these samples, the chamber atmosphere was tested for selected substances using gas detector tubes; the results are shown in Table 5. Since there are conditions under which erroneously high or low values may be obtained, Table 6 presents a list of known interferences for these tests, and the type of interference which is produced.

The following part of this section describes the results of the tests conducted for each sample material.

Sample: Y-2748 Tedlar, unprimed

The toxicity of thermodegradation products from this sample were evaluated by the MSTL procedure. The $LD_{50}$ (sample weight) for this material was 1.49 grams, with the 95% confidence interval

of 1.35 to 1.65 grams; all mortalities occurred in the chamber, thus chamber, 48 hour, and 14-day $LD_{50}$ values were the same. The TGA curve indicated decomposition began at 233.5°C and continued through 529°C; no further weight loss was observed through 712°C, although about 23.6% of the sample weight remained as a residue. Based upon this information, the tests were conducted by programming the furnace through 579°C, at 10°C/minute.

COHb levels in the dead rats ranged from 59.1 to 77.6%, with a mean of 67.8%. Detector tube analyses of the chamber atmosphere indicated 0.35 to 0.55% carbon monoxide with various sample quantities; carbon dioxide ranged from 1.5 to 1.8%; while HCN was negligable or absent (less than 5 ppm). A more detailed tabulation is presented in Table 5. Gas chromatographic (GC) analysis of the chamber atmosphere, following thermodegradation of 1.49 grams of sample (i.e., the $LD_{50}$ sample weight) taken at 3 furnace temperatures was as follows: @ 380°C carbon monoxide = 0.10%, carbon dioxide = 0.65%, & oxygen = 20.60%; @ 480°C, carbon monoxide = 0.22%, carbon dioxide = 0.98%, & oxygen = 19.65%; and @ 580°C, carbon monoxide = 0.33%, carbon dioxide = 0.88%, & oxygen = 19.76%; hydrogen cyanide was not detected.

Observations. During exposure to the pyrolysate the rats exhibited mild to severe dyspnea, mild hyperactivity, nasal discharge, ataxia, with coma and death in some animals. There were, however, no deaths during the 14-day postexposure period;

during this time, health and growth of the animals appeared normal.

Histopathology. Histological examination of tissues from animals dying in the chamber revealed the following: brain = moderate, diffuse congestion and occasional petechiae, and sometimes focal perivascular infiltrate; heart = congestion and focal, mild edema; lungs = acute mild, diffuse congestion, edema and petechiae; liver = acute mild, diffuse congestion; spleen = diffuse, moderate congestion; kidney = often normal, but sometimes acute, moderate medullary congestion; and trachea = often normal, but sometimes acute, moderate medullary congestion; and trachea = often normal, but sometimes tracheitis and diffuse mononuclear infiltrate. Histological examination of eyes, from rats dying in the chamber or sacrificed 48 hours postexposure, did not reveal any discernable abnormalities. The results from animals sacrificed 14 days after exposure were: brain = usually normal with occasional focal hemorrhage; heart = normal; lungs = mild to moderate chronic, focal pneumonitis, focal pneumonia, and atelectasis; liver = often normal, but sometimes mild, diffuse congestion; spleen = very often normal, sometimes diffuse, mild congestion; kidney = mild to moderate medullary congestion; and trachea = moderate chronic tracheitis, and focal erosion. Aniamls sacrificed 48 hours after exposure showed changes similar to those sacrificed 14 days postexposure except pneumonitis and tracheitis were less pronounced.

Sample: Y-2748B Tedlar, without adhesive

TGA analysis of this sample suggested it was essentially the same as Y-2748 (Tedlar, unprimed); subsequent animal data is also consistent with this assumption. The $LD_{50}$ for Y-2748B by the MSTL procedure was 1.43 grams, with 95% confidence interval of 1.33 to 1.54 grams. All mortalities occurred in the chamber, thus the 48 hour and 14-day $LD_{50}$ was the same. The mean COHb level was 67.2%, with a range of 55.3 to 76.4%. Gas analyses, behavior of rats during exposure, gross autopsy and histological findings were comparable to Y-2748 (Tedlar, unprimed).

Tests on this material were undertaken by the NASA procedure. After preliminary experiments to determine the appropriate sample size range, four pyrolysis experiments were conducted using 11.41, 12.55, 13.81, and 15.19 grams. The mortality pattern was 0%, 90%, 60%, and 100%, respectively. From these data, the $LD_{50}$ was approximated to be 12.55 (12.1 to 13.0) grams. More of this sample was requested so additional tests could be conducted to better pinpoint the lethality pattern. The subsequent sample of Tedlar (Y-4120) did not yield the same TGA pattern as exhibited by Y-2748 and Y-2748B (support for differences between these samples was subsequently obtained from animal exposure data), thus it was not possible to conduct additional studies on Y-2748B.

For Y-2748B, the overall mean COHb was 57.7%; breaking this down into groups it was 56.8% (range = 48.7 to 64.6%) in the group with 90% mortality, 67.1% (range = 62.0 to 79.4%) in the

group with 100% mortality. Analyses of the chamber atmosphere indicated about 0.35% CO, 2% $CO_2$, and minor amounts of HCN (up to 1.5 ppm), which was relatively constant in the range of sample weights tested.

Histological examinations of the tissues from rats that died in the chamber with the MSTL Procedure showed: brain = diffuse congestion and acute, mild edema; heart = usually normal, but sometimes with numerous petechiae; lungs = congestion with diffuse, mild acute petechiae, and atelectasis; liver = acute, diffuse mild congestion, and rare diffuse mild fatty change; spleen = very often normal, rarely acute, mild focal congestion; kidney = severe, acute medullary congestion; and trachea = some focal erosion but often normal. For those sacrificed 14-days postexposure the findings were: brain = generally normal, but some contained focal petechiae and (only) one rat had a focal perivascular mononuclear infiltrate; eyes = no abnormalities were observed; heart = no abnormalities noted; lungs = mild, focal emphysema, occasional focal atelectasis, mild focal chronic bronchitis, and mild to severe focal chronic bronchopneumonia; liver = no abnormalities noted; spleen = normal; kidney = normal; and trachea = normal.

The histopathology of organs from rats dying in the chamber for the NASA Procedure was as follows: brain = acute, mild diffuse congestion and edema and numerous petechiae; heart = generally normal, sometimes with acute, mild focal petechiae; lungs = mild to severe acute, diffuse congestion and edema, with considerable mucous in bronchioles; liver = often acute, mild to moderate

diffuse congestion; spleen = usually normal, sometimes showing perifollicular congestion; kidney = acute, mild medullary congestion; and trachea = often normal, rarely with acute/chronic tracheitis. Evaluation of organs from rats sacrificed 14-days postexposure indicated the following: brain = mild, acute focal congestion; heart = usually normal, rarely with petechiae; lungs = mild to moderate, acute, diffuse congestion and edema with increased bronchiolar secretions; liver = mild, diffuse fatty changes; spleen = diffuse, acute, moderate congestion; kidney = very often normal, rarely with mild to moderate acute medullary congestion; trachea = chronic, mild focal tracheitis.

Sample: Y-2749 Urethane Foam coated with Viton

TGA data indicated decomposition began at 218°C and was completed at 552°C, with an 8% residue at 947°C. Thus, the MSTL procedure was conducted with thermodegradation programmed through 602°C (552°C + 50°C).

The pyrolysate from this material was acutely less toxic than Tedlar, however it presented a different post-exposure pattern. Delayed deaths (i.e., those which occur after the animals were removed from the exposure chamber) were quite common in this case; they also showed a dose-dependency. In the 4 lower dose levels (2.53 through 2.95 gm), 1 rat died in the exposure chamber and there was only 1 death among the 15 survivors (6.7%); sample sizes of 3.11 and 3.27 gm produced 1 chamber death while delayed deaths accounted for 3 of the 7 survivors (42.9%); and in 5 experiments (20 rats) using 3.44 gm or more of sample, 13 rats

died in the chamber and 6 of the remaining 7 rats (85.7%) died subsequently. Thus 19 of 20 rats exposed to the pyrolysate from 3.44 grams or more of Y-2749 died; on the other hand, it required 4.21 gm or more to kill all 4 rats in the chamber.

During exposure to the Pyrolysates, the signs noted in the rats were mild to severe dyspnea, hyperactivity, ataxia, convulsions, coma, and death. Delayed deaths were common; about 40% of the total deaths occurred after the animals were removed from the exposure chamber. Some of the surviving rats exhibited signs suggestive of neurological damage, such as partial or complete paralysis of the hind legs and circular movement of head and neck toward one side (similar to that seen in inner ear disease). Another unusual post-exposure effect noted was that these rats did not grow and gain weight at the same rate as untreated rats during the 14-day period of observation. Growth retardation appeared to be directly related to the size of the pyrolyzed sample, with some of the rats that were exposed to pyrolysate from the larger samples actually losing weight. (Rats exposed to the pyrolysate of Tedlar, for example, appeared to grow normally during the 14-day post-exposure observation period).

Thermodegradation of approximately 2.5 to 4.5 gm of Urethane/Viton yielded CO levels in the same order of magnitude as seen from 1 to 2 gm of Tedlar. Unlike Tedlar, the Urethane/Viton pyrolysate consistently contained HCN. COHb levels for those rats dying in the chamber with the Urethane/Viton fumes were comparable to those dying in the Tedlar fumes: with Urethane/Viton the mean was 69.9% COHb and a range of 59.2 to 77.5%, for Tedlar these

values were 67.8% and range of 59.1 to 77.6%. While carbon monoxide may be the cause (or primary cause) of chamber deaths from both of these samples, it is quite apparent that the Urethane/Viton fumes contain some other toxicant(s), not found in Tedlar fumes which is involved in the post-exposure mortalities, neurological abnormalities, and growth suppression.

Gross autopsies following chamber deaths, delayed deaths, and after the 14-day observation period did not reveal any characteristics or lesions which were unique to exposure from this material: appearances of organs/tissues were similar to that seen from similar exposures to pyrolysates from other materials. Tissue samples were taken, fixed in bufferred formalin, sectioned, stained with hematoxylin and eosin, and evaluated histologically.

Histopathologic examination of organs from rats dying in the chamber (MSTL) revealed the following: brain = mild to moderate, diffuse congestion and edema; heart = diffuse, mild congestion and edema; lungs = acute, mild to moderate diffuse edema and congestion with numerous petechiae; liver = mild diffuse congestion with rare mild fatty change; spleen = mild, diffuse congestion; kidney = acute medullary congestion, focal petechiae and mild chronic pyelonephritis; and trachea = acute focal erosion, fresh hemorrhage, and acute/chronic tracheitis. Tissues from animals surviving the 14-day post-exposure observation period revealed the following; brain = acute, mild diffuse congestion and edema, heart = mild, focal edema and congestion with occasional petechiae;

lungs = moderate chronic focal peribronchitis, severe acute and chronic bronchopneumonia, mild focal bronchiolitis, and hyperplasia of respiratory epithelium; liver = mild diffuse congestion and moderate diffuse fatty changes; spleen = moderate perifollicular congestion; kidney = mild medullary congestion; and trachea = moderate to severe chronic tracheitis.

Sample: Y-4012 Polyimide Fabric

TGA data for the MSTL procedure indicated thermal decomposition began at 376.5°C and was completed at 513.5°C with no residue. (When conducted in an atmosphere of nitrogen, there was about a 10% residue).

The $LD_{50}$ for this sample was 0.81 gm (0.72 to 0.92 gm), with all deaths occurring in the chamber, thus this represents the chamber, 48-hour, and 14-day $LD_{50}$ values. On the basis of gross weight, this is the most toxic of all samples tested this year; however, if one calculates the $LD_{50}$ of Y-4397 (Decogard x 100) on the basis of degradable portion (based upon TGA data), the toxicity of those two samples are comparable (Y-4012 = 0.81 gm; Y-4397 = 0.85 gm). Gas analyses by detector tubes indicated a range of 0.2 to 0.4% CO in the various experiments, with HCN always present (3 to 25 ppm). Carboxyhemoglobin levels exhibited a range of 46.6 to 71.1%, with a mean of 61.0%. While some of these values are normal for carbon monoxide deaths, the trend toward lower COHb levels would suggest that HCN or some other

toxicant is exerting an effect in the lethal response.

During exposure, the rats exhibited mild to moderate hyperactivity, ataxia, muscular tremors, convulsions, coma, and death. No deaths occurred after the animals were removed from the chamber, and survivors appeared to grow normally during the 14-day post-exposure observation period.

Histopathologic examination revealed mild to severe acute diffuse congestion in the brain, heart and lungs. Some of the lungs showed acute focal hemorrhages, atelectasis, increased mucous in bronchioles, and severe bronchopneumonia, Rarely, chronic alveolar hyperplasia was noted. The liver often revealed acute, moderate, diffuse congestion. The spleen and kidneys were often normal, but occasionally showed mild to moderate acute diffuse congestion. The trachea were generally normal, but occasionally mild to moderate, acute focal erosion of mucosa was observed.

Histological examination of tissues from rats which died in the chamber (MSTL) exhibited the following: brain = mild to severe diffuse congestion and edema; heart = mild, diffuse congestion; lungs = atelectasis, and acute, severe, diffuse congestion and edema; liver = acute, diffuse congestion and acute, mild fatty changes; kidney = very often normal, sometimes with acute medullary congestion; and trachea = normal. For those animals sacrificed 14 days post-exposure, the picture was as

follows: brain = mild, diffuse congestion and edema with petechiae; heart = multiple petechiae; lungs = mild, chronic bronchopneumonia and mild to severe diffuse congestion and edema; liver = mild, diffuse fatty changes and mild, diffuse congestion; spleen = mild, acute congestion; kidney = moderate, acute medullary congestion; and trachea = usually normal, sometimes with diffuse erosion of mucosa.

Sample: Y-4120 Tedlar, deglossed

This sample was received in response to a request for an additional quantity of Tedlar unprimed or Tedlar without adhesive (Y-2748 or Y-2748B), to complete tests of the material by the NASA Procedure. Gross inspection of the sample suggested it (Y-4120) was not identical to the previous samples of Tedlar (Y-2748 & Y-2748B); this suspicion was confirmed by TGA (compare Figure 2 to Figure 16). After consultation with the Technical Monitor, it was decided to treat this as a new sample, with emphasis on the NASA Procedure.

Following preliminary experiments to determine the appropriate dose-range for this sample, 10 experiments were conducted by the NASA procedure. The results were erratic as indicated below. (See next page).

NASA Results

| Sample Weight | CO Conc. | $CO_2$ Conc. | Mortality | COHb mean (range) |
|---|---|---|---|---|
| 18.4 gm | 0.3% | 2.0% | 20% | 60.1% (59.6 - 60.6%) |
| 20.2 gm | 0.3% | 2.5% | 0% | ---------- |
| 20.2 gm | 0.3% | 2.5% | 0% | ----------(repeat) |
| 21.2 gm | 0.3% | 2.5% | 0% | ---------- |
| 22.2 gm | 0.5% | 3.0% | 100% | 69.0% (59.4 - 73.3%) |
| 23.3 gm | 0.2% | 3.0% | 0% | ---------- |
| 25.7 gm | 0.4% | 2.5% | 0% | ---------- |
| 28.2 gm | 0.4% | 3.0% | 0% | ---------- |
| 31.0 gm | 0.35% | 3.0% | 0% | ---------- |
| 24.1 gm | 0.5% | 2.5% | 80% | 58.8% (44.8 - 77.0%) |

One of the obvious problems encountered in the above tests was that of sample volume. Because of the relatively low toxicity of the fumes generated and low density of the sample, the volume of the required sample sizes exceeded the design capacity of the pyrolysis tube, thus it was necessary to wrap the sample rather tightly around the moveable rod which led to incomplete and inconsistent thermal degradation of the test sample. This could have been the cause of the inconsistent mortality pattern shown above. There was not enough sample left to conduct additional tests of this type. The samll quantity of sample which remained was employed in the MSTL Procedure to ascertain if its level of

toxicity was comparable to that observed earlier for Y-2748 and Y-2748B.

MSTL Procedure. The $LD_{50}$ weight for Y-2748 was chosen as the initial dose level, i.e., 1.49 gm. No deaths occurred, either in the chamber or during the 14-day post-exposure observation period. Other tests using 1.65 gm and 2.24 gm did not produce any mortalities, and 3.03 gm of the sample killed only one exposed rat. The lack of sample prevented any further tests by this method. However these data do indicate thermodegradation products from this sample (Y-4120) were less toxic to exposed rats than the other Tedlar samples (Y-2748 and Y-2748B). The rat which died from the largest sample tested by this method had a COHb value of 65.4%, suggesting that carbon monoxide was very probably the cause of death. There were no delayed deaths in any of the animals exposed to fumes from this material.

Histological data for this material is not as complete as for the other samples, however the information does not suggest qualitatively different lesions than observed for most other materials. The rats dying in the NASA chamber indicated the following: brain = mild, acute, diffuse congestion and edema; heart = mild, acute, diffuse congestion and focal petechiae; liver = mild to moderate, acute, diffuse congestion, with atelectasis and acute focal hemorrhage; liver = mild to severe, acute, diffuse congestion; spleen = normal; and kidney = mild to moderate, acute medullary congestion. Rats sacrificed 14 days after exposure by

the MSTL procedure showed the following: brain = moderate, acute, diffuse congestion and edema; heart = focal petechiae; lungs = focal atelectasis, mild, acute, diffuse congestion and edema; liver = mild, diffuse congestion; spleen = mild, diffuse congestion; kidney = normal; and trachea = mild, focal chronic inflammation.

Sample: Y-4157 Kel-F on Nylon

Data from the TGA curve for the MSTL procedure indicated that thermal degradation of this sample occurred between 213°C and 583°C, with about 65% (by weight) of the sample being decomposed. The pyrolysis furnace, therefore, was programmed through 635°C. The weight of this sample required to kill 50% of the exposed rats was 5.51 gm (4.78 to 6.35 gm) for chamber deaths, and slightly less for 48 hour and 14 day observations, 5.15 gm (4.57 to 5.79 gm).

The carboxyhemoglobin levels in rats dying in the chamber ranged from 57.7 to 69.6%, with a mean of 62.2%. Carbon monoxide levels in the chamber were found to range from approximately 0.2 to 0.6%, and HCN (10 to >60 ppm) was consistently observed.

During exposure, the rats exhibited hyperactivity, mild to severe dyspnea, ataxia, occasional convulsions, coma and death.

Histological examination of organs from rats dying in the chamber showed the following: brain = normal; heart = normal; lungs = mild, acute, focal hemorrhage, congestion and edema, with atelectasis; liver = massive acute congestion; spleen = normal; kidney = acute medullary congestion; and trachea = normal. Animals

sacrificed 14 days after exposure revealed: brain = focal petechiae; heart = normal; lungs = mild, chronic, focal bronchopneumonia, and mild, focal edema; liver = severe, focal hemorrhages and diffuse, acute fatty changes; spleen = normal; kidney = normal; and trachea = hyperplasia of peritracheal node.

Sample: Y-4158 Nomex Fabric, Red

The furnace was programmed up to 570°C for thermal decomposition of this sample in the MSTL procedure. TGA data indicated that about 7.25% of this sample was not volatilized, but degradation was initiated at a rather low temperature (175°C). The chamber $LD_{50}$ was 1.27 gm (1.20 to 1.34 gm) while the 48-hour and 14-day $LD_{50}$ values were only slightly lower, 1.26 gm (1.19 to 1.33 gm). Due to variations in mortality pattern of individual runs, each sample weight was run in triplicate, thus the $LD_{50}$ is based upon 12 rat exposures/sample weight.

The mean COHb level in rats dying within the chamber was 60.9%, with a range of 51.5 to 81.3%; CO levels in the chamber atmosphere ranged from 0.2 to 0.35%. HCN was consistently present, ranging from 3 to 18 ppm. During exposure the rats exhibited hyperactivity, mild to severe dyspnea,ataxia, occasional convulsions, coma, and death.

Histological examination of tissues from the rats dying in the (MSTL) chamber showed the following: brain = mild, acute, diffuse congestion and edema, with multiple petechiae; heart = often normal, sometimes with mild, acute, diffuse congestion;

lungs = focal hemorrhage, alveolar hyperplasia, and interstitial pneumonitis; liver = mild to severe, acute, diffuse congestion; spleen = often normal, sometimes mild, acute, diffuse congestion; kidney = moderate, acute medullary congestion; and trachea = very often normal, sometimes acute, focal erosion. Examination of tissues from those animals which survived 14 days post-exposure revealed: brain = often normal, some with meningeal hemorrhage; heart = normal; lungs = acute and chronic bronchitis and massive, acute bronchopneumonia; liver = acute, diffuse congestion; spleen = often normal, sometimes mild, acute congestion; kidney = often normal, sometimes mild to severe acute, diffuse congestion; and trachea = normal.

Sample: Y-4159 50% Wool-50% Leavil Fabric

The TGA experiments indicated this material began to decompose at about 215°C, with rapid degradation in which 50% of the sample (by weight) pyrolyzed under 300°C, and complete volatilization by 527°C. For these studies (MSTL procedure) the furnace was programmed up through 600°C. The $LD_{50}$ of this sample was 2.35 gm (2.13 to 2.58 gm) for chamber deaths, and 2.18 gm (2.03 to 2.35 gm) for both 48-hour and 14-day deaths.

Carboxyhemoglobin levels of rats expiring in the chamber strongly suggest the acute mortalities are not due solely to carbon monoxide. The mean COHb was 48%, with a range of 38.9 to 56.6%. Based upon the composition of this sample, one would anticipate cyanide and chloride to be present. Tests for HCN and $Cl_2$ indicated these gases were present, but in far smaller quantities than expected if these are to be considered the lethal component(s) of the fumes from the material. It is possible that

the maximum concentration of these substances was produced early in the pyrolysis process, and by the time the chamber atmosphere was analyzed the substance may have been largely 'washed out' of the chamber by the continuous air flow. Another plausible explanation relates to the form in which the cyanide and chloride exist; the nature of these compounds may be such that they are quite toxic biologically, but are not detected as HCN or $Cl_2$. The alternative to these postulates is that cyanide and chloride do not exist in the pyrolysate -- this is deemed unlikely from a wool/PVC material, but if true, then there must be some other toxic substance present.

During exposure to the fumes of this material the rats exhibited hyperactivity, mild to severe dyspnea, nasal and oral secretions, ataxia, strong abdominal muscular movements, convulsions, coma, and death.

Histopathological examination of tissues from animals dying in the chamber revealed: brain = mild, acute, diffuse congestion and edema; heart = mild, acute, diffuse congestion and edema; lungs = mild to moderate, acute bronchopneumonia and moderate, diffuse atelectasis; liver = mild, acute, diffuse congestion; spleen = normal; kidney = mild, acute, diffuse medullary congestion; and trachea = often normal, sometimes focal erosion of mucosa. Similar examination of the 14-day survivors showed: brain = mild, acute, diffuse edema and congestion with focal petechiae; heart = mild, acute, focal congestion; lungs = focal

chronic bronchopneumonia, increased mucous, moderate, focal alveolar hyperplasia, chronic bronchitis, and mild, acute focal congestion; liver = mild, acute, diffuse congestion; spleen = normal; kidney = mild, acute medullary congestion; and trachea = very often normal, occasionally chronic tracheitis.

Sample: Y-4397 Decogard x 100

TGA data on this sample indicates it has a rather high thermodegradation temperature, with decomposition occurring between 507 and 628°C for the MSTL conditions, and 537 and 775°C for the NASA conditions; in both cases there was a large residue of 82 and 84%, respectively.

For the MSTL procedure, the furnace was programmed up to 680°C. The weight of sample required to kill 50% of the exposed rats was 4.71 gm (4.27 to 5.20 gm). Since all deaths occurred in the chamber, this represents the $LD_{50}$ for all 3 time periods (chamber, 48-hour and 14-days). Based upon TGA data, this would represent degradation of 0.85 gm as the $LD_{50}$.

The mean COHb level of the dead rats was 61.3%, and a range of 48.4 to 78.5%. Carbon monoxide concentrations, as determined by gas detector tube, ranged from 0.2 to 0.5%, and HCN from 5 to 30 ppm. All deaths from the pyrolysate of this material by the NASA procedure also occurred in the chamber. The chamber, 48-hour, and 14-day $LD_{50}$ values were identical for the NASA procedure, and this was 8.64 gm (7.81 to 9.56 gm). Based on the TGA information of 84% residue, then this would represent degradation of 1.38 gm as the $LD_{50}$. COHb levels of dead rats

ranged from 56.6 to 76.5%, with a mean of 63.3%, which is quite comparable to that obtained by the MSTL procedure.

During exposure to the fumes, the rats exhibited hyperactivity, mild to severe dyspnea, occasional ataxia and muscle tremors, convulsions, coma, and death.

The histopathologic picture shows considerable similarity between tissues of rats exposed by the MSTL procedure to those exposed by the NASA procedure, in which the lungs show the greatest reaction to the pyrolysate with the reaction tending to be somewhat more severe (in intensity) by the NASA procedure. Evaluation of tissues from rats dying in the chamber by the MSTL procedure; brain = normal; heart = normal; lungs = mild, acute, diffuse congestion and edema, mild, focal atelectasis, massive acute inflammation and abscesses; liver = normal; spleen = normal; kidney = mild to moderate, acute medullary congestion; and trachea = normal. The 14-day survivors from the MSTL procedure showed: brain = normal; heart = normal; lungs = mild, focal chronic bronchopneumonia, moderate, diffuse chronic vasculitis, and massive abscesses; liver = mild to moderate, diffuse chronic congestion; spleen = megakaryocytes; kidney = normal; and trachea = normal. Chamber deaths of rats by the NASA procedure revealed: brain = often normal, sometimes mild to moderate, acute diffuse congestion and edema with numerous petechiae; heart = normal; lungs = mild to severe, diffuse acute congestion and edema, mild, focal atelectasis, and hemorrhage; liver = mild, diffuse acute congestion and moderate, diffuse fatty changes; spleen = normal;

kidney = mild to moderate, acute medullary congestion; and trachea = moderate, acute tracheitis. Tissues from the 14-day post-exposure survivors of the NASA procedure showed the following: brain = normal; heart = usually normal, rarely numerous petechiae; lungs = massive abscesses, emphysema, mild to severe, acute, diffuse congestion, chronic pneumonitis and bronchopneumonia; liver = normal; spleen = normal; kidney = normal; and trachea = rarely moderate, diffuse chronic tracheitis.

Figure 32 depicts the relationship between the concentration of carbon monoxide and hydrogen cyanide as a function of the weight of sample pyrolyzed by the NASA procedure.

## IV. Discussion

A few comments concerning the similarities and differences between the NASA and MSTL procedures might be appropriate.

Both systems employ thermal degradation (by an electric furnace) of the test sample and exposure of rats to the fumes generated. The NASA confines all fumes, produced by relatively rapid degradation, in the exposure chamber. Thus the qualitative and quantitative nature of the fumes should be the same throughout the time of exposure of the animals, varying only in regard to (1) unstable species which may undergo spontaneous conversion to another compound(s), (2) removal of certain substances from the atmosphere by the animals, or (3) addition of certain substances (e.g., $CO_2$) to the atmosphere from the animals. On the other hand, the MSTL procedure is a dynamic system in which air is continually flowing through the exposure chamber, coupled with a slower heating rate, may result in a peak concentration of one

toxicant occurring at a different time than the peak concentration of another toxicant. Therefore if carbon monoxide is produced at a lower temperature than hydrogen cyanide, or vice versa, then the animals would be exposed to peak concentrations of these sequentially rather than simultaneously. For this reason, analysis of the chamber atmosphere for the MSTL procedure is more time-dependent (in terms of determining maximum concentration of the substance in question) than the NASA procedure.

Typically, the results to date have indicated most materials to have a greater absolute toxicity (although not necessarily a greater relative toxicity) by the MSTL procedure than by the NASA procedure. This could be due to one or a combination of the following factors: (1) the smaller chamber size in the MSTL procedure, (2) possibly higher peaks of toxicant(s) to which the rats are exposed, (3) the longer period of exposure for the MSTL procedure, and/or (4) the slower heating rate, and possibly the more oxygen-rich environment during thermodegradation.

These comments are not presented as an argument for or against either of the techniques, but rather to point out some of the differences which should be kept in mind when comparing or interpreting the results obtained by these two procedures.

One of the more unique and interesting samples tested was the Polyurethane and Viton; particularly because of (1) the relatively large number of delayed deaths which resulted, (2) the apparent neurological dysfunction, and (3) the retardation of weight gain in rats exposed to the fumes of this material. These were not observed in animals exposed to fumes of the other materials evaluated.

The Tedlar material(s) may also deserve a comment. It appears that we had two different materials, one was coded Y-4120 and the other coded Y-2748 and Y-2748B. The pyrolysis-toxicity data indicate Y-4120 (Tedlar, deglossed) produced considerably less toxic fumes than was produced by Y-2748 & Y-2748B (Tedlar, unprimed & Tedlar, without adhesive). The reason for the differences in TGA and toxicity of fumes is not known; it could be a difference in primary formulation, or it could be a coating or other type of additives, for Y-2748 & Y-2748B, placed on the basic formulation (represented by Y-4120).

Two samples which may have more in common than would appear at first glance are Y-4012 (Polyimide fabric) and Y-4397 (Decogard x 100). The $LD_{50}$ for Y-4012 was 0.81 gm by the MSTL procedure, while that for Y-4397 was 4.71 gm by the same procedure. TGA results of Y-4397 indicated an 18% weight loss (decomposition) by temperatures up to 1,000°C, with shiny, needle-like strands remaining. The information we received about this sample indicated it was a polyimide resin on fiberglas. The polyimide fabric (Y-4012) decomposed completely during TGA test. If one makes the assumption that all of the polyimide in Decogard is pyrolyzed, with fiberglas remaining as the residue, and adjust the $LD_{50}$ accordingly (i.e., 4.71 gm x 18% = 0.85 gm), then there is remarkably good agreement between the $LD_{50}$ values for these two samples in terms of toxicity of pyrolysis products.

One of the characteristics of both of these test methods is that they produce information concerning the relative toxicity of the materials tested under the conditions of the specific test. While the quantity of sample needed to kill 50% of the exposed animals by the respective procedures is generally less for the MSTL procedure than the NASA procedure, no constant factor has been found to date which will convert data from one procedure into comparable values for the other procedure. Therefore the data tends to assume quantitative significance only when compared to other materials evaluated under the same set of conditions. In order to better visualize the potential toxicity of thermodegradation products from the NASA samples tested, Table 7 is included to provide general information of the levels of toxicity produced by various other materials when tested by the MSTL procedure. As additional information is obtained from the NASA samples, one will have a better idea of the general range of toxicity of the various types of materials when thermally decomposed, and may gain a better appreciation of the direction and magnitude of changes in toxicity when the material is modified (see, for example, Y-2748 & Y-2748B Tedlar vs. Y-4120 Tedlar).

Table 1

IDENTIFICATION OF SAMPLES RECEIVED FOR EVALUATION

| Code | Description of Sample | Procedure Employed |
|---|---|---|
| Y-2748 | Tedlar, unprimed, JSC # 3-13-4 | MSTL |
| Y-2748B | Tedlar (without adhesive) | MSTL<br>NASA |
| Y-2749 | Urethane Foam coated with Viton (A.D. Little # 151B) | MSTL |
| Y-4012 | Polyimide Fabric (gold color) | MSTL |
| Y-4120 | Tedlar, deglossed | NASA<br>MSTL |
| Y-4157 | Kel-F 2401 on Nylon (fabric) | MSTL |
| Y-4158 | Nomex Upholstery Fabric, Anchorage II Red 69/1895 | MSTL |
| Y-4159 | Fabric, 50% Wool/50% Leavil (PVC) Zirconium Treated, Style ML-8965-48, Intercel-Langenthal | MSTL |
| Y-4397 | Decogard X 100 (Polyimide Resin on Fiberglas) | MSTL<br>NASA |

Table 2

RESULTS OF THERMOGRAVIMETRIC ANALYSIS (TGA) STUDIES

| Sample # | Y-2748 | Y-2748 | Y-2748 | Y-2748B* | Y-2748B |
|---|---|---|---|---|---|
| Identification | Tedlar, unprimed | Tedlar, unprimed | Tedlar, unprimed | Tedlar (without adhesive) | Tedlar (without adhesive) |
| TGA Run No. | 153 | 161 | 165 | 180 & 181 | 204 |
| Atmosphere | Air | Air | Nitrogen | Air | Air |
| Flow Rate | 200 ml/min | 20 ml/min | 20 ml/min | 20 ml/min | 200 ml/min |
| Heating Rate | 10°C/min | 20°C/min | 20°C/min | 20°C/min | 10°C/min |
| Sample Wt. | 10.15 mg | 10.22 mg | 9.57 mg | 2.04 mg | 1.98 mg |
| Initiation of Decomposition | 233.5°C | 225.3°C | 241.6°C | 213.6°C | 218.8°C |
| Completion of Decomposition | 529.3°C | 524.7°C | 592.3°C | 560.7°C | 528.2°C |
| Maximum TGA Temp. | 712°C | 994.3°C | 1002.5°C | 931.25°C | 1019.3°C |
| Final Residue Wt. | 2.4 mg | 2.48 mg | 2.34 mg | 0.52 mg | 0.48 mg |
| Percent Final Residue | 23.64% | 24.3% | 24.5% | 25.77% | 24% |
| Temperature for 50% Degradation | 403.0°C | 390.0°C | 421.8°C | 399.2°C | 596.8°C |
| Percent Residue at 600°C | 23.64% | 24.3% | 24.5% | 25.77% | 24% |

*Mean values of duplicate determinations.

Table 2 (cont'd)

RESULTS OF THERMOGRAVIMETRIC ANALYSIS (TGA) STUDIES

| Sample # | Y-2749 | Y-2749 | Y-2749 | Y-2749 | Y-2749 |
|---|---|---|---|---|---|
| Identification | Urethane & Viton | Urethane & Viton | Urethane & Viton | Urethane & Viton | Urethane & Viton |
| TGA Run No. | 159 | 163 | 158 | 167 | 169 |
| Atmosphere | Air | Air | Nitrogen | Nitrogen | Helium |
| Flow Rate | 200 ml/min | 20 ml/min | 200 ml/min | 20 ml/min | 200 ml/min |
| Heating Rate | 10°C/min | 20°C/min | 10°C/min | 20°C/min | 10°C/min |
| Sample Wt. | 10.46 mg | 10.49 mg | 10.40 mg | 9.74 mg | 10.50 mg |
| Initiation of Decomposition | 218.3°C | 201.7°C | 231.2°C | 223.5°C | 243.0°C |
| Completion of Decomposition | 551.6°C | 529.3°C | 735.7°C | 567.5°C | 645.8°C |
| Maximum TGA Temp. | 947.3°C | 894.0°C | 860.0°C | 966.3°C | 960.8°C |
| Final Residue Wt. | 0.84 mg | 0.70 mg | 1.43 mg | 1.09 mg | 0.98 mg |
| Percent Final Residue | 8.03% | 6.7% | 13.75% | 11.2% | 9.3% |
| Temperature for 50% Degradation | 381.9°C | 396.5°C | 429.4°C | 419.5°C | 426.0°C |
| Percent Residue at 600°C | 8.03% | 6.7% | 15.67% | 11.2% | 12.8% |

*Mean values of duplicate determinations.

Table 2 (cont'd)

RESULTS OF THERMOGRAVIMETRIC ANALYSIS (TGA) STUDIES

| Sample # | Y-4012 | Y-4012* | Y-4012 | Y-4012 | Y-4012* |
|---|---|---|---|---|---|
| Identification | Polyimide Fabric | Polyimide Fabric | Polyimide Fabric | Polyimide Fabric | Polyimide Fabric |
| TGA Run No. | 174 | 175-B & 176 | 175-A | 177 | 178 & 179 |
| Atmosphere | Air | Air | Nitrogen | Helium | Helium |
| Flow Rate | 200 ml/min | 20 ml/min | 200 ml/min | 200 ml/min | 20 ml/min |
| Heating Rate | 10°C/min | 20°C/min | 10°C/min | 10°C/min | 20°C/min |
| Sample Wt. | 9.84 mg | 1.99 mg | 10.02 mg | 9.98 mg | 1.99 mg |
| Initiation of Decomposition | 376.5°C | 395.8°C | 427.6°C | 442.6°C | 414.0°C |
| Completion of Decomposition | 513.5°C | 608.9°C | Incomplete 1045.3°C | Incomplete 1048.3°C | 825.2°C |
| Maximum TGA Temp. | 678.2°C | 702.9°C | 1045.3°C | 1048.3°C | 922.4°C |
| Final Residue Wt. | 0.0 mg | 0.0 mg | 1.04 mg | 1.00 mg | 0.0 mg |
| Percent Final Residue | 0.0% | 0.0% | 10.37% | 10.02% | 0.0% |
| Temperature for 50% Degradation | 498.0°C | 558.3°C | 663.0°C | 860.0°C | 653.3°C |
| Percent Residue at 600°C | 0.0% | 8.03% | 73% | 75% | 68.5% |

*Mean values of duplicate determinations.

Table 2 (cont'd)

RESULTS OF THERMOGRAVIMETRIC ANALYSIS (TGA) STUDIES

| Sample # | Y-4120* | Y-4120* | Y-4157* | Y-4157* | Y-4157* |
|---|---|---|---|---|---|
| Identification | Tedlar,deglossed | Tedlar,deglossed | Kel-F on Nylon | Kel-F on Nylon | Kel-F on Nylon |
| TGA Run No. | 182 & 183 | 303 & 304 | 189 & 190 | 309 & 310 | 217 & 219 |
| Atmosphere | Air | Air | Air | Nitrogen | Helium |
| Flow Rate | 20 ml/min | 200 ml/min | 200 ml/min | 200 ml/min | 200 ml/min |
| Heating Rate | 20°C/min | 10°C/min | 10°C/min | 10°C/min | 10°C/min |
| Sample Wt. | 2.03 mg | 2.00 mg | 2.00 mg | 2.02 mg | 1.99 mg |
| Initiation of Decomposition | 240.6°C | 249.2°C | 212.9°C | 212.9°C | 216.5°C |
| Completion of Decomposition | 557.5°C | 541.5°C | 583.3°C | 1004.6°C | 767.6°C |
| Maximum TGA Temp. | 838.5°C | 811.0°C | 1012.6°C | 1004.6°C | 767.6°C |
| Final Residue Wt. | 0.0 mg | 0 mg | 0.69 mg | 0.87 mg | 0.93 mg |
| Percent Final Residue | 0.0% | 0% | 34.5% | 43.1% | 47% |
| Temperature for 50% Degradation | 386.6°C | 384.9°C | 457.1°C | 752.6°C | 680.6°C |
| Percent Residue at 600°C | 0.0% | 0% | 34.5% | 53.8% | 53% |

*Mean values of duplicate determinations.

Table 2 (cont'd)

RESULTS OF THERMOGRAVIMETRIC ANALYSIS (TGA) STUDIES

| Sample # | Y-4158* | Y-4158* | Y-4158 | Y-4159* | Y-4159* |
|---|---|---|---|---|---|
| Identification | Nomex Fabric | Nomex Fabric | Nomex Fabric | Wool/Leavil Fabric | Wool/Leavil Fabric |
| TGA Run No. | 186, 187, 188 | 311 & 312 | 218 | 184 & 185 | 305 & 306 |
| Atmosphere | Air | Nitrogen | Helium | Air | Nitrogen |
| Flow Rate | 200 ml/min | 200 ml/min | 200 ml/min | 200 ml/min | 200 ml/min |
| Heating Rate | 10°C/min | 10°C/min | 10°C/min | 10°C/min | 10°C/min |
| Sample Wt. | 2.05 mg | 2.03 mg | 2.08 mg | 2.02 mg | 2.12 mg |
| Initiation of Decomposition | 174.0°C | 177.6°C | 40.8°C | 214.7°C | 211.3°C |
| Completion of Decomposition | 515.2°C | 1021.7°C | 714.4°C | 527.0°C | 838.3°C |
| Maximum TGA Temp. | 752.6°C | 1021.7°C | 878.3°C | 778.6°C | 838.3°C |
| Final Residue Wt. | 0.15 mg | 0.70 mg | 0.12 mg | 0.0 mg | 0.12 mg |
| Percent Final Residue | 7.25% | 34.5% | 6% | 0.0% | 5.7% |
| Temperature for 50% Degradation | 495.8°C | 624.3°C | 572.0°C | 281.0°C | 317.2°C |
| Percent Residue at 600°C | 7.25% | 51.0% | 50% | 0.0% | 11.3% |

*Mean values of duplicate determinations.

Table 2 (cont'd)

RESULTS OF THERMOGRAVIMETRIC ANALYSIS (TGA) STUDIES

| Sample # | Y-4397* | Y-4397* | Y-4397* | | |
|---|---|---|---|---|---|
| Identification | Decogard x 100 | Decogard x 100 | Decogard x 100 | | |
| TGA Run No. | 220 & 221 | 301 & 302 | 307 & 308 | | |
| Atmosphere | Air | Air | Nitrogen | | |
| Flow Rate | 200 ml/min | 20 ml/min | 200 ml/min | | |
| Heating Rate | 10°C/min | 20°C/min | 10°C/min | | |
| Sample Wt. | 2.04 mg | 2.17 mg | 2.07 mg | | |
| Initiation of Decomposition | 507.3°C | 537.1°C | 518.5°C | | |
| Completion of Decomposition | 628.2°C | 775.2°C | 734.6°C | | |
| Maximum TGA Temp. | 1001.1°C | 945.9°C | 1014.3°C | | |
| Final Residue Wt. | 1.67 mg | 1.83 mg | 1.77 mg | | |
| Percent Final Residue | 82% | 84.3% | 85.5% | | |
| Temperature for 50% Degradation | 561.8°C** | 609.3°C | 633.2°C | | |
| Percent Residue at 600°C | 85% | 89.9% | 94.7% | | |

*Mean values of duplicate determinations.

**This is a derived value based upon two assumptions: (1) the fiberglas portion is completely stable to these temperatures, and (2) that the polyimide portion of the sample is completely degraded at these temperatures. Thus this would represent the temperature required to thermally degrade 50% of the polyimide.

Table 3

WEIGHT OF SAMPLE REQUIRED TO KILL 50% OF RATS ($LD_{50}$)
WHEN THERMALLY DEGRADED ACCORDING TO PROTOCOL

| Code | Sample | Duration of Observation | $LD_{50}$ (95% Confidence Interval) in grams | Test Protocol |
|---|---|---|---|---|
| Y-2748 | Tedlar, unprimed | Chamber Deaths | 1.49 (1.35 to 1.65) | MSTL |
| | | 48 hour Deaths | 1.49 (1.35 to 1.65) | MSTL |
| | | 14 day Deaths | 1.49 (1.35 to 1.65) | MSTL |
| Y-2748B | Tedlar, without adhesive | Chamber Deaths | 1.43 (1.33 to 1.54) | MSTL |
| | | 48 hour Deaths | 1.43 (1.33 to 1.54) | MSTL |
| | | 14 day Deaths | 1.43 (1.33 to 1.54) | MSTL |
| Y-2748B | Tedlar, without adhesive | Chamber Deaths | 12.55 (12.1 to 13.0)* | NASA |
| | | 48 hour Deaths | 12.55 (12.1 to 13.0)* | NASA |
| | | 14 day Deaths | 12.55 (12.1 to 13.0)* | NASA |
| Y-2749 | Urethane Foam coated with Viton | Chamber Deaths | 3.65 (3.35 to 3.81) | MSTL |
| | | 48 hour Deaths | 3.19 (3.00 to 3.38) | MSTL |
| | | 14 day Deaths | 3.15 (2.96 to 3.34) | MSTL |
| Y-4012 | Polyimide Fabric | Chamber Deaths | 0.81 (0.72 to 0.92) | MSTL |
| | | 48 hour Deaths | 0.81 (0.72 to 0.92) | MSTL |
| | | 14 day Deaths | 0.81 (0.72 to 0.92) | MSTL |
| Y-4120 | Tedlar, deglossed | | > 3.0 gm.** | MSTL |

*Note! This is an approximation of the $LD_{50}$. After preliminary experiments, there was only enough sample to conduct 4 experiments; these gave mortalities of 0%, 90%, 60% and 100%. The approximation was based upon these data.

**Most of this sample was consumed in the NASA procedure tests, and thus there was not enough sample to complete the $LD_{50}$ determination. However, sample weights up through 2.2 gm. were thermally degraded without killing any of the exposed rats, and only 1 rat died from 3.0 gm, and no delayed deaths.

Table 3 (continued)

| Code | Sample | Duration of Observation | $LD_{50}$ (95% Confidence Interval) in grams | Test Protocol |
|---|---|---|---|---|
| Y-4120 | Tedlar, deglossed | | >31.0 gm.*** | NASA |
| Y-4157 | Kel-F on Nylon | Chamber Deaths | 5.51 (4.78 to 6.35) | MSTL |
| | | 48 hour Deaths | 5.15 (4.57 to 5.79) | MSTL |
| | | 14 day Deaths | 5.15 (4.57 to 5.79) | MSTL |
| Y-4158 | Nomex Fabric | Chamber Deaths | 1.27 (1.20 to 1.34) | MSTL |
| | | 48 hour Deaths | 1.26 (1.19 to 1.33) | MSTL |
| | | 14 day Deaths | 1.26 (1.19 to 1.33) | MSTL |
| Y-4159 | Fabric, 50% Wool/50% Leavil | Chamber Deaths | 2.35 (2.13 to 2.58) | MSTL |
| | | 48 hour Deaths | 2.18 (2.03 to 2.35) | MSTL |
| | | 14 day Deaths | 2.18 (2.03 to 2.35) | MSTL |
| Y-4397 | Decogard X 100 | Chamber Deaths | 4.71 (4.27 to 5.20) | MSTL |
| | | 48 hour Deaths | 4.71 (4.27 to 5.20) | MSTL |
| | | 14 day Deaths | 4.71 (4.27 to 5.20) | MSTL |
| Y-4397 | Decogard X 100 | Chamber Deaths | 8.64 (7.81 to 9.56) | NASA |
| | | 48 hour Deaths | 8.64 (7.81 to 9.56) | NASA |
| | | 14 day Deaths | 8.64 (7.81 to 9.56) | NASA |

***The relatively low toxicity of thermodegradation products from this sample required the use of large quantities per test which created problems of sample bulk. Results were somewhat erratic: 22.3 gm. sample killed 10/10 (100%) of exposed rats, however none of the 30 rats exposed to 20.2 to 21.2 gm samples died; 34.1 gm killed 80% of exposed rats, but none of the 40 rats exposed to 23.3 to 31.0 gm samples died. The supply of this sample was exhausted before further tests could be conducted to resolve the questions concerning the observed results.

Table 4

CO & COHb Data from MSTL Procedure

| Sample | | COHb Levels of Rats Dying in Chamber (mean & range) | $LD_{50}$ Sample Weight | Temperature when Gas Sample Taken | GC Determination of CO Concentration in Chamber Atmosphere |
|---|---|---|---|---|---|
| Y-2748 | Tedlar, unprimed | 67.8% (59.1 - 77.6%) | 1.49 gm | 380°C<br>480°C<br>580°C | 0.10%<br>0.22%<br>0.33% |
| Y-2749 | Urethane/ Viton | 69.9% (59.2 - 77.5%) | 3.65 gm | 402°C<br>502°C<br>602°C | 0.10%<br>0.42%<br>0.31% |
| Y-4012 | Polyimide Fabric | 61.0% (46.6 - 71.1%) | 0.81 gm | 380°C<br>480°C<br>580°C | 0.07%<br>0.07%<br>0.22% |
| Y-4120 | Tedlar, deglossed | 65.4% ( only 1 rat ) | 3.03 gm* | 410°C<br>510°C<br>610°C | 0.0%<br>0.2%<br>0.2% |
| Y-4157 | Kel-F on Nylon | 62.2% (57.7 - 69.6%) | 5.51 gm | 435°C<br>535°C<br>635°C | 0.11%<br>0.17%<br>0.13% |
| Y-4158 | Nomex Fabric | 60.9% (51.5 - 81.3%) | 1.27 gm | 370°C<br>470°C<br>570°C | 0.00%<br>0.27%<br>0.18% |
| Y-4159 | Wool/Leavil | 48.0% (38.9 - 56.5%) | 2.35 gm | 400°C<br>500°C<br>600°C | 0.00%<br>0.10%<br>0.10% |

*This is not the $LD_{50}$ weight of sample. Because of sample limitation the $LD_{50}$ could not be determined; 3.03 gm was the largest sample tested by this procedure and it killed 1 of the 4 rats, lesser quantities of sample tested did not kill any rats.

Table 4 (continued)

| Sample | COHb Levels of Rats Dying in Chamber (mean & range) | $LD_{50}$ Sample Weight | Temperature when Gas Sample Taken | GC Determination of CO Concentration in Chamber Atmosphere |
|---|---|---|---|---|
| Y-4397 Decogard | 61.3% (48.4 - 78.5%) | 4.71 gm** | 480°C<br>580°C<br>680°C | 0.10%<br>0.20%<br>0.16% |

**TGA data indicated only 18% (by weight) of this sample decomposed at the maximum temperature of this experiment. Extrapolation based upon this would indicate degradation of 0.85 gm of the sample would have occurred for the $LD_{50}$. If this is the polyimide degrading, it compares quite favorably to an $LD_{50}$ of 0.81 gm for the polyimide fabric (Y-4012).

Table 5

Gases Detected* and Approximate Concentration Range in Pyrolysates from NASA Materials

| Sample | Method | $CO_2$% | CO% | HCN ppm | $Cl_2$ ppm | Acetone% | $PH_3$ ppm | $H_2S$ ppm | $SO_2$% | NO + $NO_2$ ppm |
|---|---|---|---|---|---|---|---|---|---|---|
| Y-2748 | MSTL | 1.5 - 1.8 | 0.35 — 0.55 | <5 | N.D. | 0.035 | N.D. | N.D. | N.D. | N.D. |
| Y-2748B | MSTL | 1.0 - 1.75 | 0.3 — 0.5 | (-) | (-) | 0.02-0.07 | (-) | (-) | (-) | (-) |
| Y-2748B | NASA | 2.0 | 0.35 | <5 | (-) | (-) | (-) | (-) | (-) | (-) |
| Y-2749 | MSTL | 0.5 - 2.0 | 0.1 — 0.6 | 1-12 | N.D. | 0.02-0.07 | N.D. | N.D. | N.D. | N.D. |
| Y-4012 | MSTL | 1.0 - 2.0 | 0.2 — 0.4 | 3-25 | (-) | (-) | (-) | (-) | (-) | (-) |
| Y-4120 | MSTL | 1.5 | 0.2 — 0.3 | (-) | (-) | 0.1 | (-) | (-) | (-) | (-) |
| Y-4120 | NASA | 2.0 - 3.0 | 0.2 — 0.5 | N.D. | (-) | 0.1 — 0.2 | (-) | (-) | (N.D.) | (-) |
| Y-4157 | MSTL | 1.5 - 2.5 | 0.2 — 0.6 | 10-60 | N.D. | 0.05-0.2 | N.D. | N.D. | N.D. | N.D. |
| Y-4158 | MSTL | 1.0 - 1.5 | 0.2 — 0.35 | 3-18 | N.D. | <0.01 | N.D. | N.D. | N.D. | N.D. |
| Y-4159 | MSTL | 1.0 - 1.8 | 0.1 — 0.3 | 2-10 | <1 | 0.01—0.02 | N.D. | N.D. | N.D. | N.D. |
| Y-4397 | MSTL | 1.0 - 1.5 | 0.2 — 0.5 | 5-30 | N.D. | N.D. | N.D. | N.D. | N.D. | N.D. |
| Y-4397 | NASA | 2.5 - >3.0 | 0.3 — 0.5 | 5-12 | N.D. | 0.3 | (-) | <100 | <0.5 | (-) |

* Results from Bendix/Gastec or Bendix/Kitagawa gas detector tubes.
N.D. = not detected by the gas detector tube.
(-) = test not conducted for this sample.

Table 6

Sensitivity Range and Reported Interfering Gases for Gas Detector Tubes

| Desired Test | Sensitivity Range* | Reported Interfering Gases | Type of Interences |
|---|---|---|---|
| Carbon Dioxide ($CO_2$) | 0.2 to 3.0% | $SO_2$, $NO_2$, HCN, HCL if >10% of $CO_2$ conc.<br>$H_2S$, $Cl_2$ if >5% of $CO_2$ | Excess concentrations produce similar stain |
| Carbon Monoxide (CO) | 0.2 to 5.0% | Propane < 20%; Butane < 10% Hexane <0.3%; Acetylene <0.3%; Ethylene <0.5%; Alcohols, Esters, Ketones, Ethers, Aromatics ... all up to a few % | These are eliminated in the precleaning layer, thus show no effect except in higher concentrations. |
| Hydrogen Cyanide (HCN) | 5 to 60 ppm | $SO_2$, $H_2S$ <5 ppm have no effect<br>HCL, $HNO_3$, $H_2SO_4$, $Cl_2$ | If >5 ppm produce similar stain.<br>No effect, eliminated in precleaning area. These produce pink stain in precleaning area which is proportional to concentration. If entire precleaning area is discolored, these give a false positive test. |

*Higher or lower levels than the stated range may be estimated by using multiple strokes of the gas sampling pump, or by using fractional parts of the standard (100 cc.)stroke, and adjusting the reading accordingly.

Table 6 (cont'd)

| Desired Test | Sensitivity Range* | Reported Interfering Gases | Type of Interference |
|---|---|---|---|
| Chlorine ($Cl_2$) | 1 to 30 ppm | Halogens, $O_3$, $NO_2$ | Same stain, thus false positive reading. |
| Acetone | 0.01 to 0.8% | Methane, Ethane, Ethylene Propane | No effect |
| | | | Discolors entire reagent dark brown at 0.2% |
| | | Acetylene | Discolors entire reagent dark brown at 0.3% |
| | | Other Organic Vapors | Produce similar stains by themselves |
| Phosphine ($PH_3$) | 50 to 500 ppm | $H_2S$ >20% of $PH_3$; $H_2Se$ >33% of $PH_3$; $AsH_3$ >20% of $PH_3$ | All yield a positive reaction |
| Hydrogen Sulfide ($H_2S$) | 100 to 1600 ppm | $SO_2$ >1/15 of $H_2S$ conc. | Erroneously high reading |
| Sulfur Dioxide ($SO_2$) | 0.5 to 4.0% | $H_2S$ >500 ppm | Produces brown stain |
| | | $H_2S$ >2% of $SO_2$ conc. | Erroneously high reading if coexisting (orange to green stain) |
| Oxides of Nitrogen ($NO + NO_2$) | 25 to 600 ppm | Halogens and Chlorine dioxide | Erroneously high reading if present; produces same stain; |
| | | $SO_2$ >2000 ppm; $H_2S$ >1000 ppm | No stain by itself, but may produce a minus error. |

*Higher or lower levels than the stated range may be estimated by using multiple strokes of the gas sampling pump, or by using fractional parts of the standard (100 cc.) stroke, and adjusting the reading accordingly.

TABLE 7

PYROLYSIS TOXICITY OF A VARIETY OF MATERIALS, MSTL PROCEDURE

| Identification | $LD_{50}$* gm. | 95% Confidence Interval, gm. | Average Relative % COHb ± Standard Deviation | Gases Detected Besides CO and $CO_2$ |
|---|---|---|---|---|
| UNTREATED COTTON FABRICS | | | | |
| Flannel | 1.97 | 1.85 to 2.12 | 69.9 ± 4.4 | *** |
| Corduroy | 4.23 | 2.63 to 6.80 | 75.3 ± 4.4 | $H_2S$; $Cl_2$ and $SO_2$, trace |
| Brown Fabric | 4.25 | 3.39 to 5.36 | 73.2 ± 3.9 | *** |
| Blue Fabric | 6.41 | 6.19 to 6.66 | 67.1 ± 1.3 | $Cl_2$, trace |
| Yellow Fabric | 4.12 | 3.64 to 4.68 | 63.9 ± 11.5 | HCN, trace |
| TREATED COTTON FABRICS | | | | |
| Flannel, FR** | 1.61 | 1.56 to 1.68 | 69.6 ± 6.0 | HCN, trace |
| Knit, FR | 1.59 | 1.57 to 1.62 | 70.0 ± 3.2 | *** |
| Knit, FR | 1.47 | 1.44 to 1.51 | 70.9 ± 5.4 | *** |
| Fabric, FR | 3.21 | 3.02 to 3.41 | 74.5 ± 2.6 | HCN |
| Fabric, FR | 2.31 | 2.02 to 2.67 | 76.5 ± 0.3 | HCN, trace |
| Knit, FR | 1.71 | 1.65 to 1.79 | 69.5 ± 5.2 | *** |
| Twill | 2.40 | 2.32 to 2.50 | 67.9 ± 3.9 | *** |
| Orange-red Fabric | 2.63 | 1.79 to 3.90 | 67.1 ± 2.0 | HCN, trace |
| Durable-Press Fabric | 1.98 | 1.90 to 2.06 | 67.7 ± 2.8 | HCN |
| Fabric, FR | 1.54 | 1.51 to 1.57 | 78.4 ± 6.0 | HCN |
| Fabric, FR | 2.31 | 2.25 to 2.38 | 74.4 ± 8.1 | HCN |
| Fabric, FR | 2.23 | 1.98 to 2.52 | 59.0 ± 11.5 | HCN |

*$LD_{50}$ and 95% confidence intervals were not calculated by Karber's method, however the values are comparable to those obtained by use of Karber's method.

**FR = Flame Retardant-Treated Material

***No Gas Analyses Conducted

TABLE 7 (continued)

| Identification | $LD_{50}$ gm. | 95% Confidence Interval, gm. | Average Relative % COHb ± Standard Deviation | Gases Detected Besides CO and $CO_2$ |
|---|---|---|---|---|
| NONCOTTON FABRICS | | | | |
| Acrylic Knit | 1.59 | 1.33 to 1.91 | 20.5 ± 14.3 | HCN |
| Polyester Knit | 2.00 | 1.98 to 2.04 | 73.8 ± 2.4 | *** |
| Fortrel-Cotton Blend | 2.31 | 2.21 to 2.42 | 67.0 ± 4.4 | *** |
| Nylon Knit | 4.57 | 3.99 to 5.24 | 71.5 ± 1.8 | HCN |
| Rayon Velvet | 9.84 | 8.20 to 11.81 | 67.9 ± 4.2 | *** |
| Napped Nylon | 5.23 | 5.07 to 5.39 | 71.7 ± 3.6 | HCN, trace |
| Polyester | 1.75 | 1.66 to 1.85 | 70.0 ± 4.6 | *** |
| Acetate | 3.43 | 3.07 to 3.84 | 70.7 ± 5.7 | *** |
| Wool | 0.36 | 0.35 to 0.38 | 20.7 ± 4.1 | HCN |
| Wool | 1.32 | 1.22 to 1.41 | 31.3 ± 5.3 | HCN |
| Nomex (NASA) | 0.33 | 0.24 to 0.47 | 43.8 ± 10.9 | HCN; $Cl_2$ trace |
| Durette (NASA) | 0.19 | 0.17 to 0.21 | 46.7 ± 18.0 | HCN |
| Modified Nomex HT-4 (NASA) | 0.14 | 0.14 to 0.15 | 17.2 ± 2.7 | HCN |
| YARNS | | | | |
| Wool | 0.30 | 0.27 to 0.35 | 36.1 ± 6.3 | HCN; $H_2S$; $SO_2$ trace |
| Orlon | 0.27 | 0.24 to 0.33 | 8.8 ± 13.0 | HCN |
| Wool-Nylon Blend | 1.73 | 0.97 to 3.08 | 65.1 ± 5.7 | HCN |
| Wool | 0.28 | 0.22 to 0.39 | 46.2 ± 1.1 | HCN |
| Wool | 0.52 | 0.44 to 0.63 | 45.8 ± 7.5 | HCN,trace |
| Wool | 0.32 | 0.26 to 0.41 | 43.7 ± 3.4 | HCN |

TABLE 7 (continued)

| Identification | $LD_{50}$ gm. | 95% Confidence Interval, gm. | Average Relative % COHb ± Standard Deviation | Gases Detected Besides CO and $CO_2$ |
|---|---|---|---|---|
| POLYURETHANE FOAMS | | | | |
| Flexible | 3.64 | 3.00 to 4.42 | 59.2 ± 9.6 | HCN, trace |
| Flexible | 3.13 | 2.56 to 3.83 | 72.2# | HCN, trace |
| Flexible | 1.87 | 1.74 to 2.01 | 70.2 ± 1.8 | HCN |
| Flexible | 3.61 | 3.51 to 3.73 | 59.4 ± 2.5 | HCN |
| Flexible | 1.84 | 1.53 to 2.23 | 61.2 ± 19.0 | HCN; $Cl_2$, trace |
| Urethane Foam (NASA) | 0.78 | 0.62 to 0.97 | 76.0 ± 5.4 | HCN |

#Only one analysis is available

Figure 1

TGA Run No. 153

Y-2748 Tedlar, unprimed

Atmosphere: Air
Flow Rate: 200 ml/min
Heating Rate: 10°C/min
Sample Weight: 10.15 mg

23.64% Residue @ 600°
50% Residue @ 403.0°C
23.64% Residue @ 712.0°C

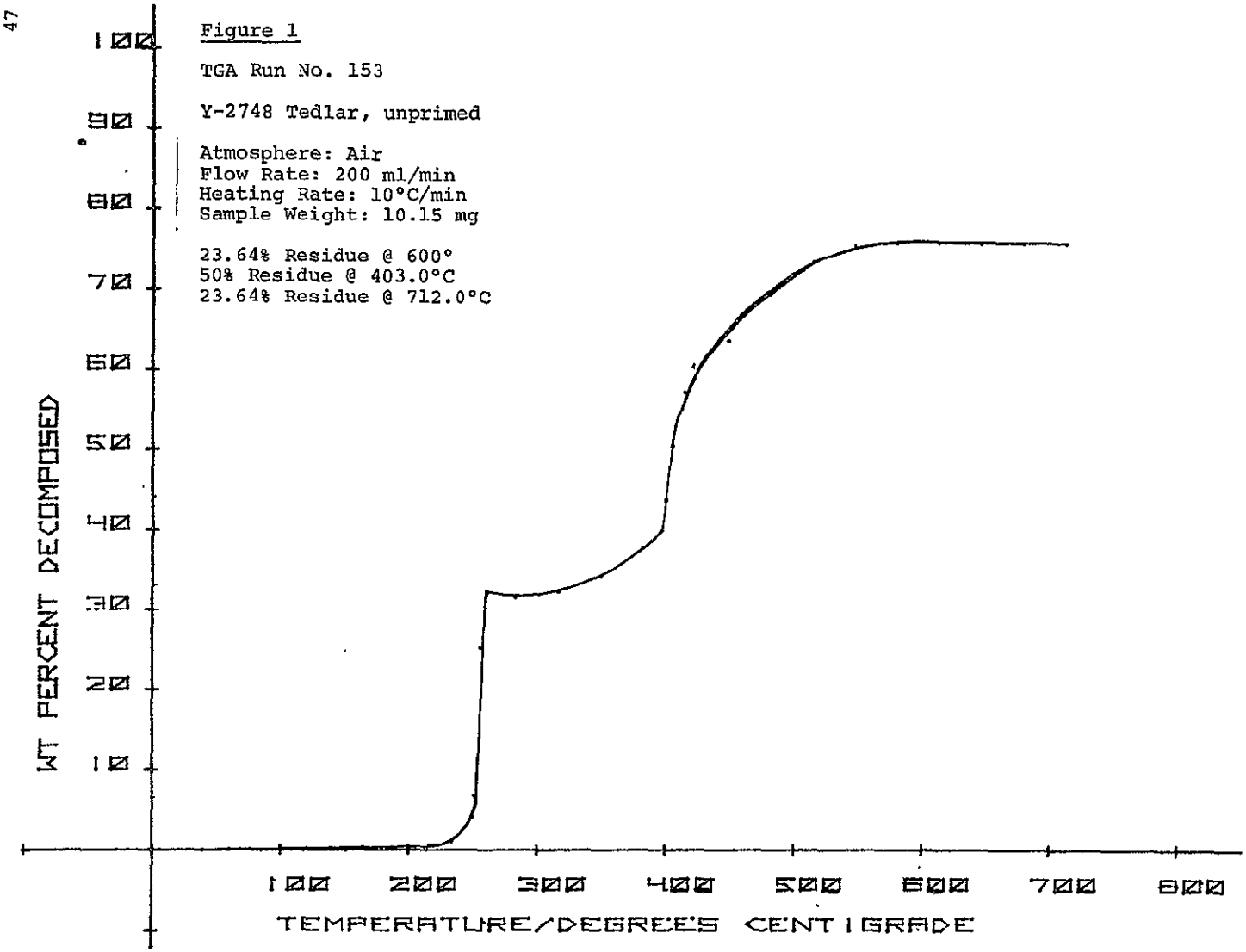


Figure 2

TGA Run No. 161

Y-2748 Tedlar, unprimed

Atmosphere: Air
Flow Rate: 20 ml/min
Heating Rate: 20°C/min
Sample Weight: 10.22 mg

24.3% Residue @ 600°C
50% Residue @ 390°C
24.3% Residue @ 994.3°C

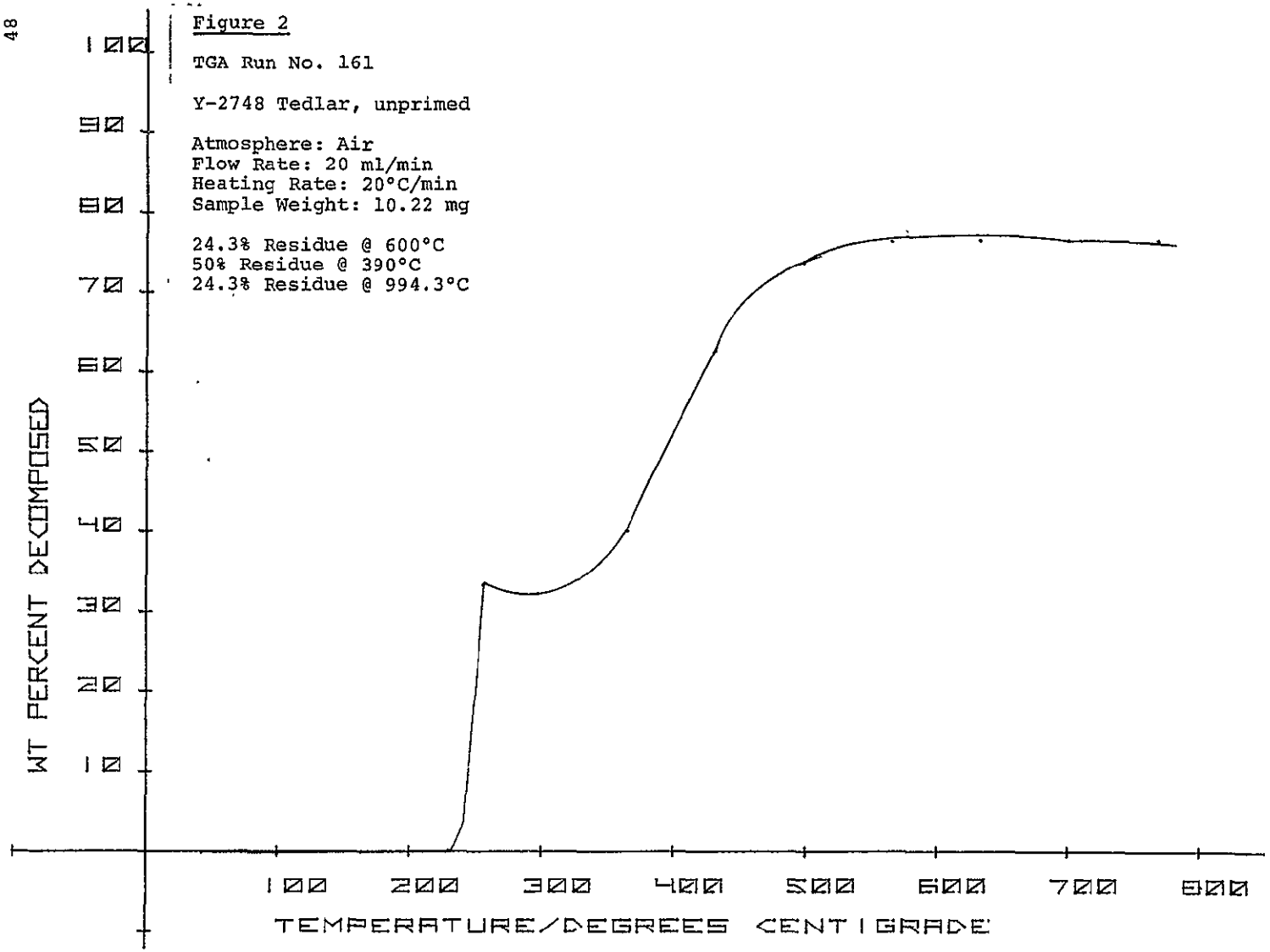


Figure 3

TGA Run No. 165

Y-2748 Tedlar, unprimed

Atmosphere: Nitrogen
Flow Rate: 20 ml/min
Heating Rate: 20°C/min
Sample Weight: 9.57 mg

24.5% Residue @ 600°C
50% Residue @ 421.8°C
24.5% Residue @ 1002.5°C

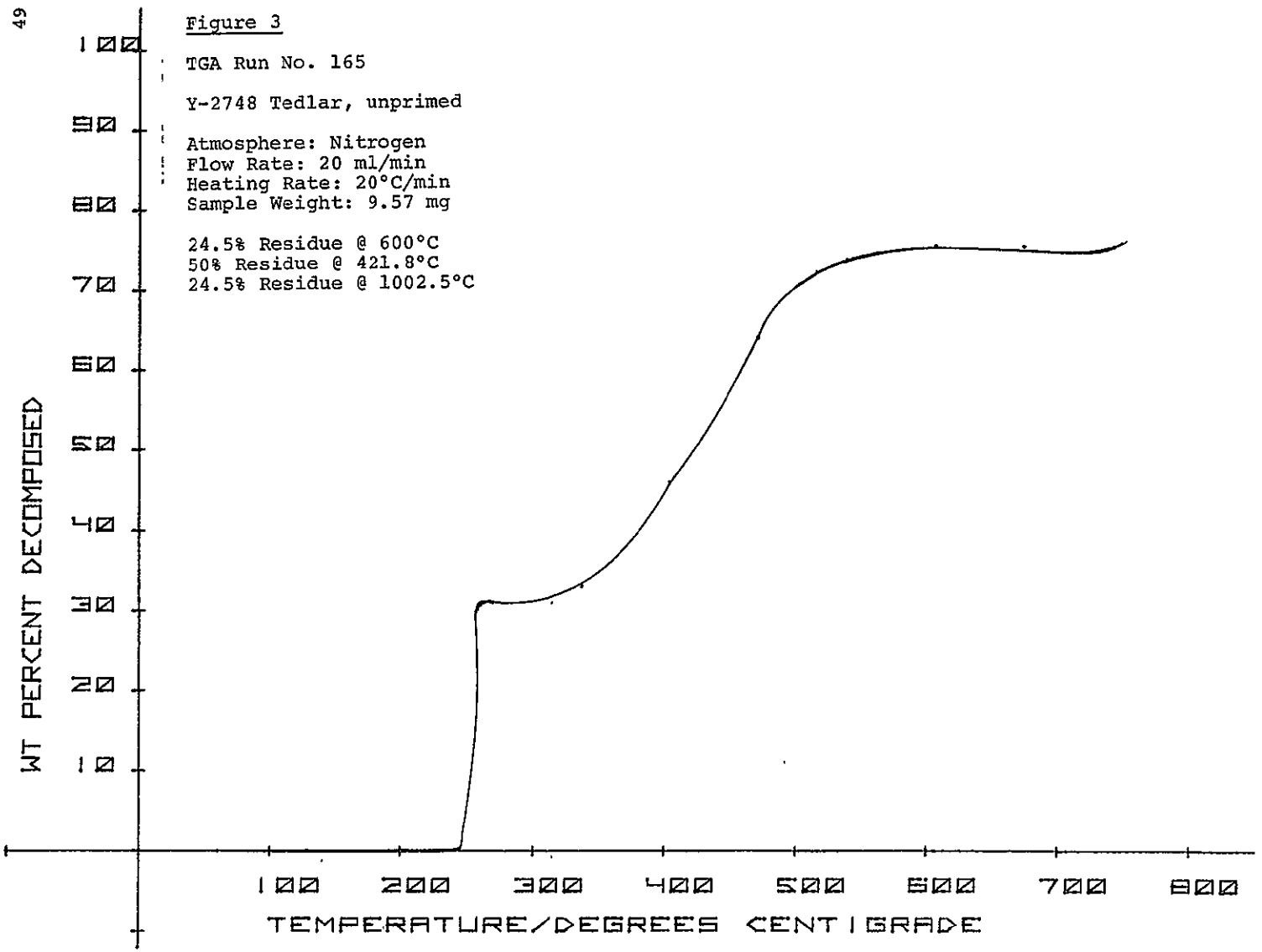


Figure 4

TGA Run No.181

Y-2748B Tedlar(without adhesive)

Atmosphere: Air
Flow Rate: 20 ml/min
Heating Rate: 20°C/min
Sample Weight: 2.04 mg

25.77% Residue @ 600°C
50% Residue @ 399.2°C
25.77% Residue @ 931.25°C

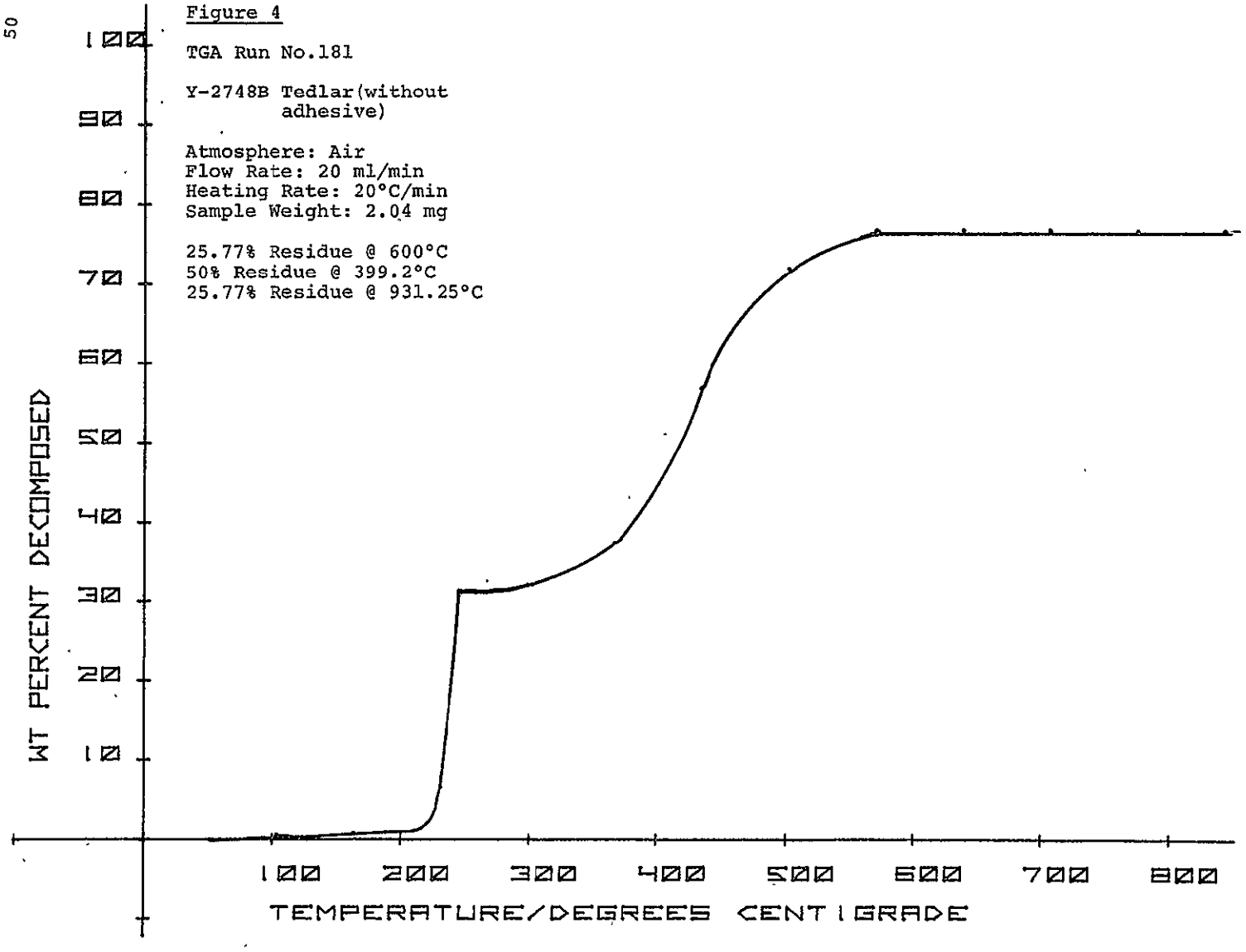


Figure 5

TGA Run No. 204

Y-2748B Tedlar(without adhesive)

Atmosphere: Air
Flow Rate: 200 ml/min
Heating Rate: 10°C/min
Sample Weight: 1.98 mg

24% Residue @ 600°C
50% Residue @ 596.8°C
24% Residue @ 1019.3°C

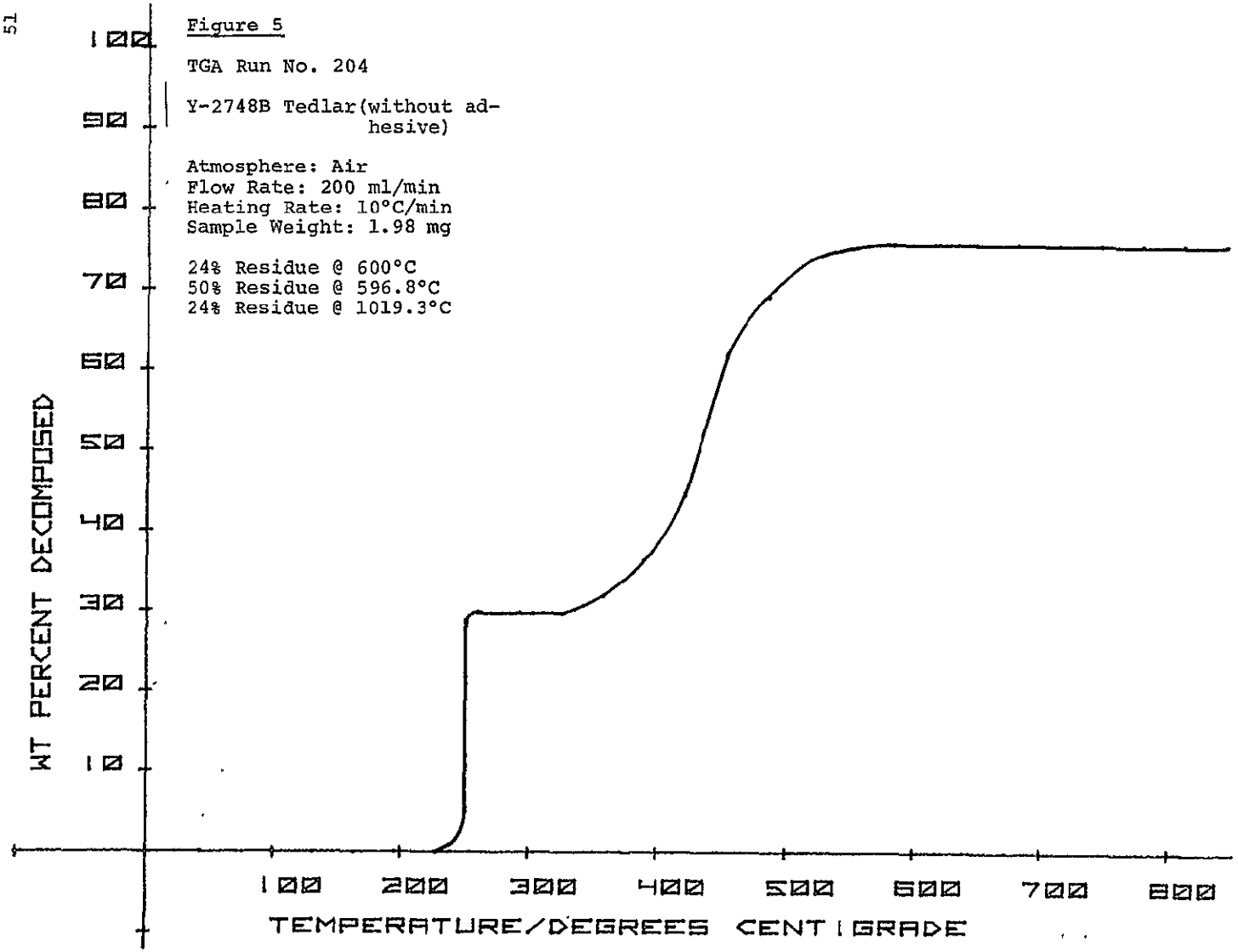


Figure 6

TGA Run No. 159

Y-2749 Urethane & Viton

Atmosphere: Air
Flow Rate: 200 ml/min
Heating Rate: 10°C/min
Sample Weight: 10.46 mg

8.03% Residue @ 600°C
50% Residue @ 381.9°C
8.03% Residue @ 947.3°C

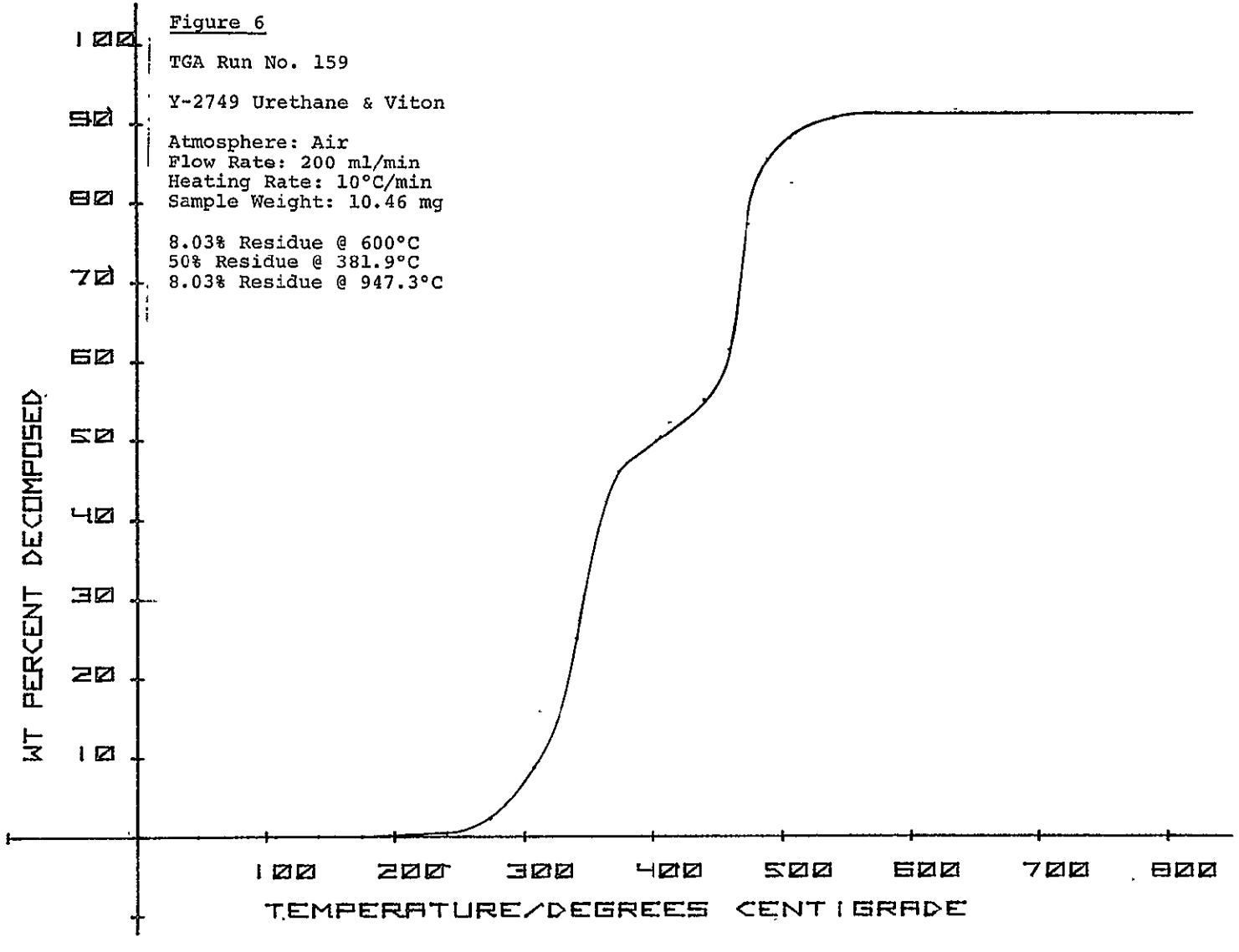


Figure 7

TGA Run No. 163

Y-2749 Urethane & Viton

Atmosphere: Air
Flow Rate: 20 ml/min
Heating Rate: 20°C
Sample Weight: 10.49 mg

6.7% Residue @ 600°C
50% Residue @ 396.5°C
6.7% Residue @ 894.0°C

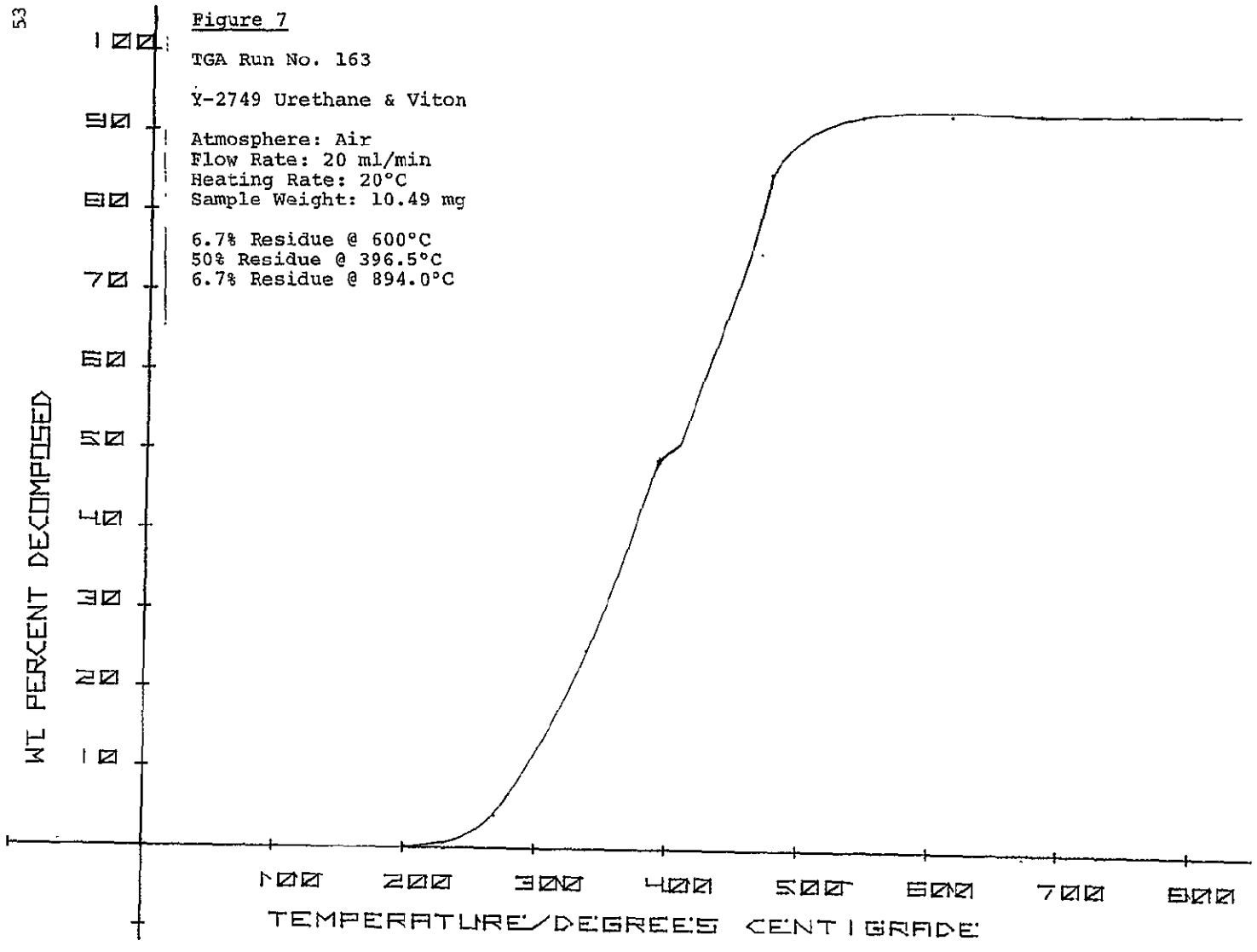


Figure 8

TGA Run No. 158

Y-2749 Urethane & Viton

Atmosphere: Nitrogen
Flow Rate: 200 ml/min
Heating Rate: 10°C/min
Sample Weight: 10.40 mg

15.67% Residue @ 600°C
50% Residue @ 429.4°C
13.75% Residue @ 860.0°C

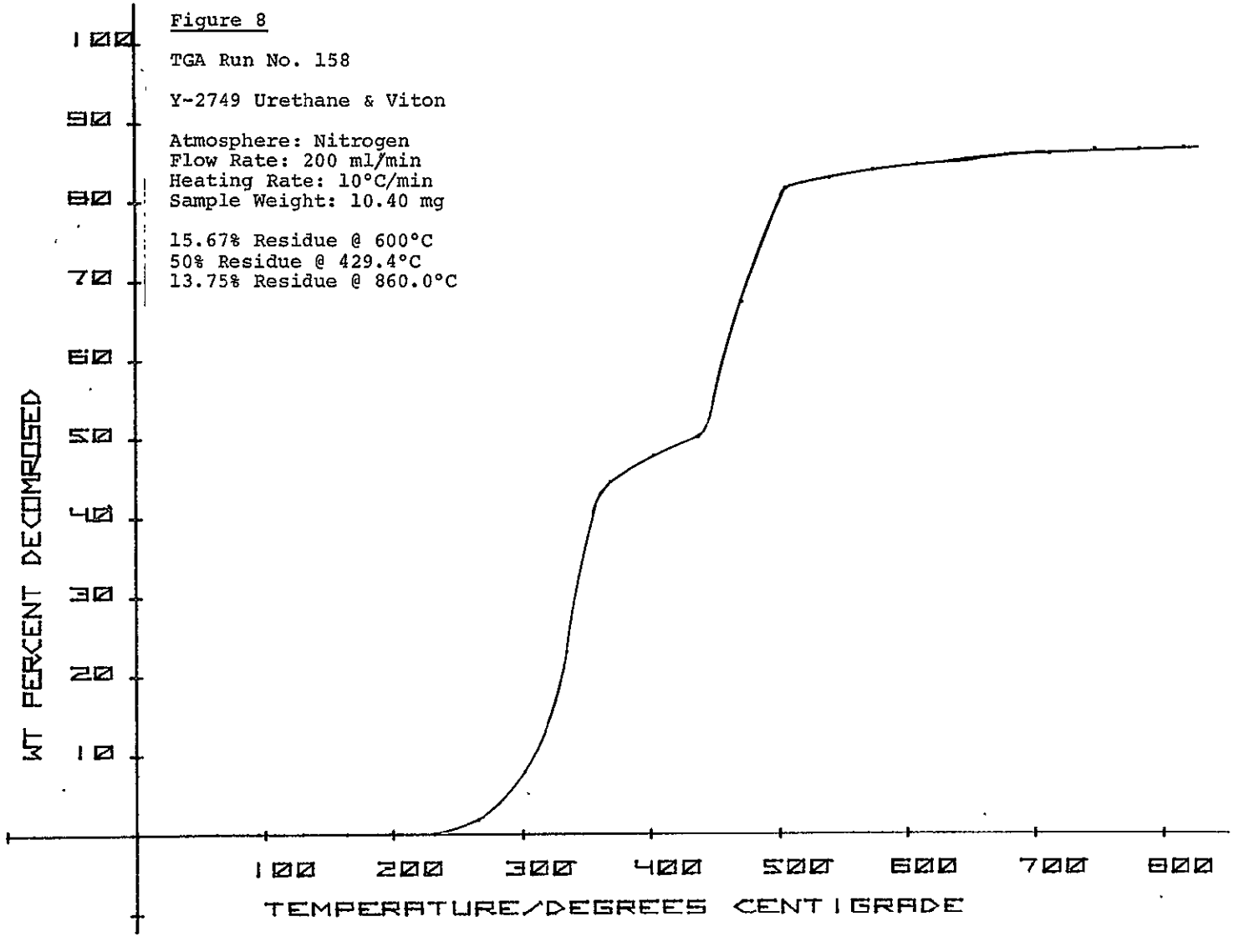


Figure 9

TGA Run No. 167

Y-2749 Urethane & Viton

Atmosphere: Nitrogen
Flow Rate: 20 ml/min
Heating Rate: 20°C/min
Sample Weight: 9.74 mg

11.2% Residue @ 600°C
50% Residue @ 419.5°C
11.2% Residue @ 966.3°C

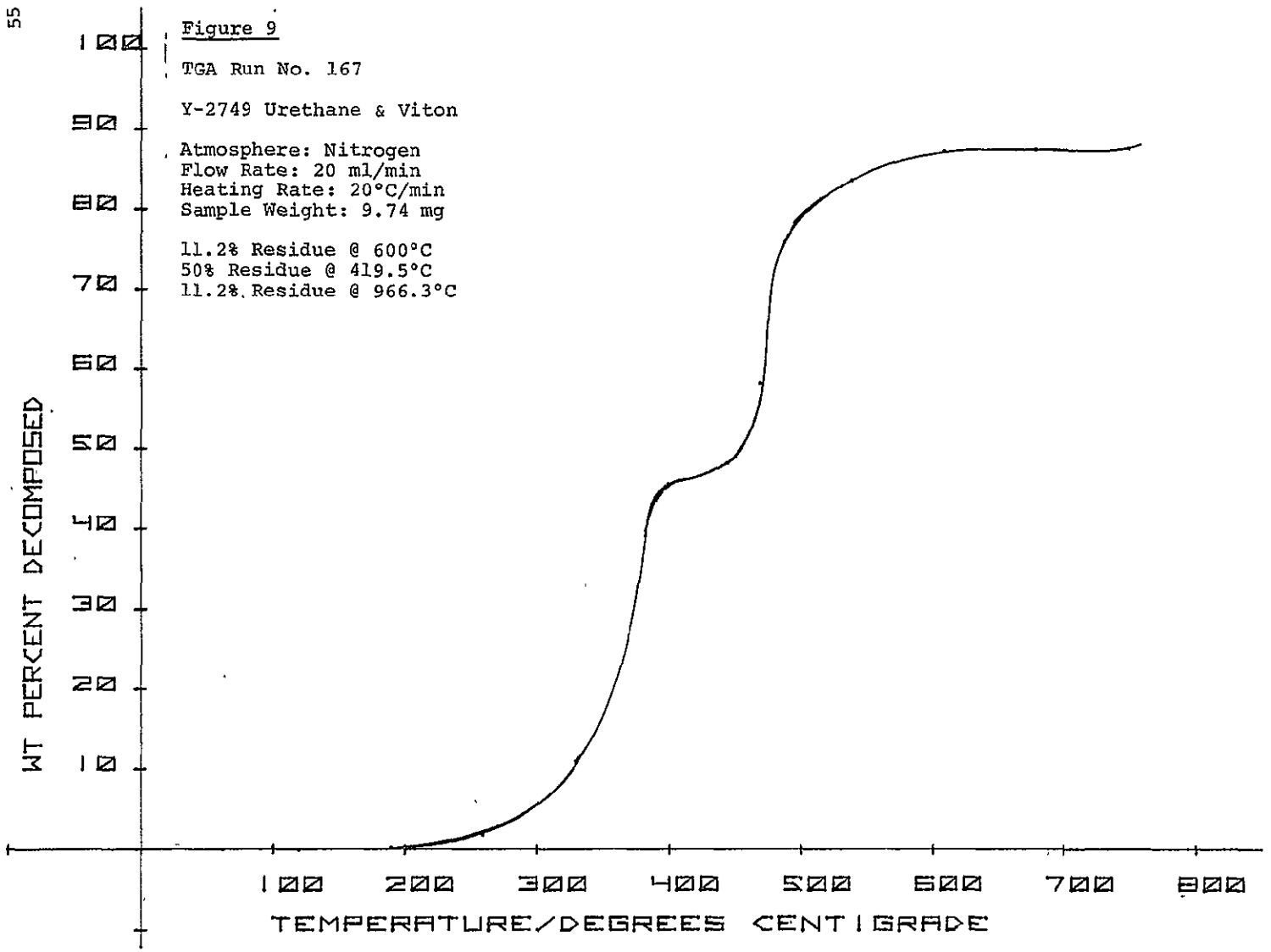


Figure 10

TGA Run No. 169

Y-2749 Urethane & Viton

Atmosphere: Helium
Flow Rate: 200 ml/min
Heating Rate: 10°C/min
Sample Weight: 10.50 mg

12.8% Residue @ 600°C
50% Residue @ 426.0°C
9.3% Residue @ 960.8°C

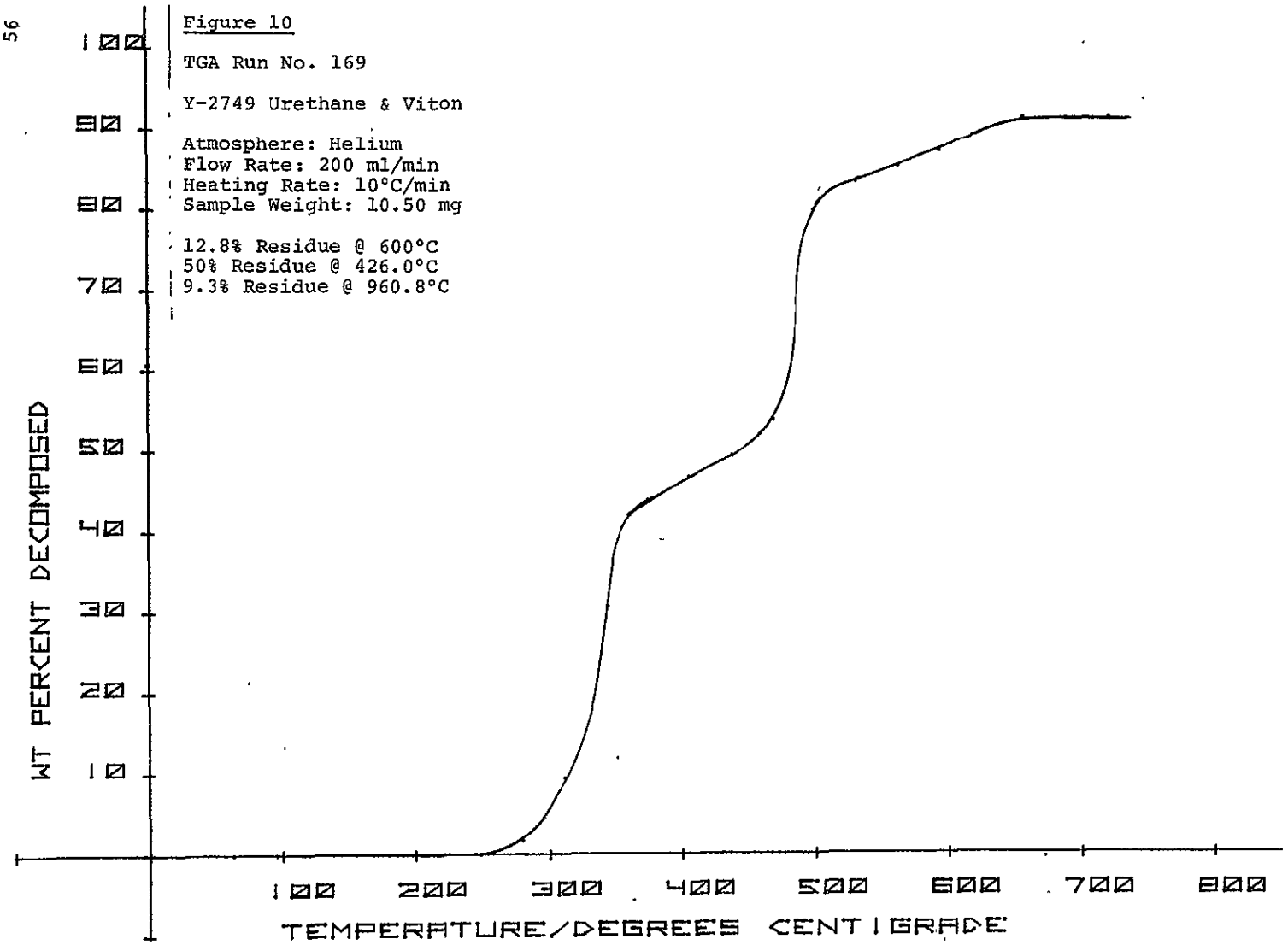


Figure 11

TGA Run No. 174

Y-4012 Polyimide Fabric

Atmosphere: Air
Flow Rate: 200 ml/min
Heating Rate: 10°C/min
Sample Weight: 9.84 mg

0.0% Residue @ 600°C
50% Residue @ 498.0°C
0.0% Residue @ 678.2°C

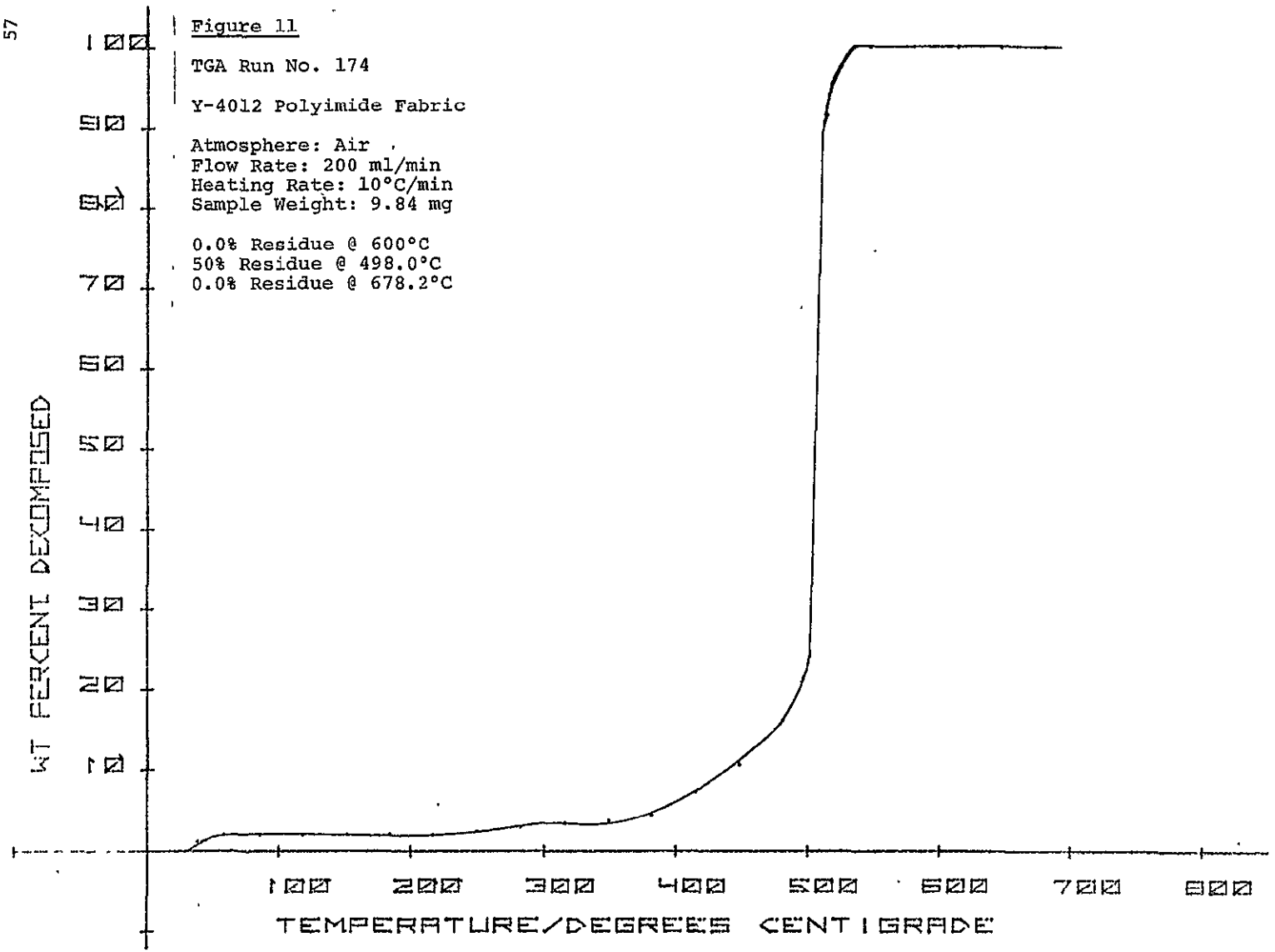


Figure 12

TGA Run No. 175-B

Y-4012 Polyimide Fabric

Atmosphere: Air
Flow Rate: 20 ml/min
Heating Rate: 20°C/min
Sample Weight: 1.99 mg

8.03% Residue @ 600°C
50% Residue @ 558.3°C
0.0% Residue @ 702.9°C

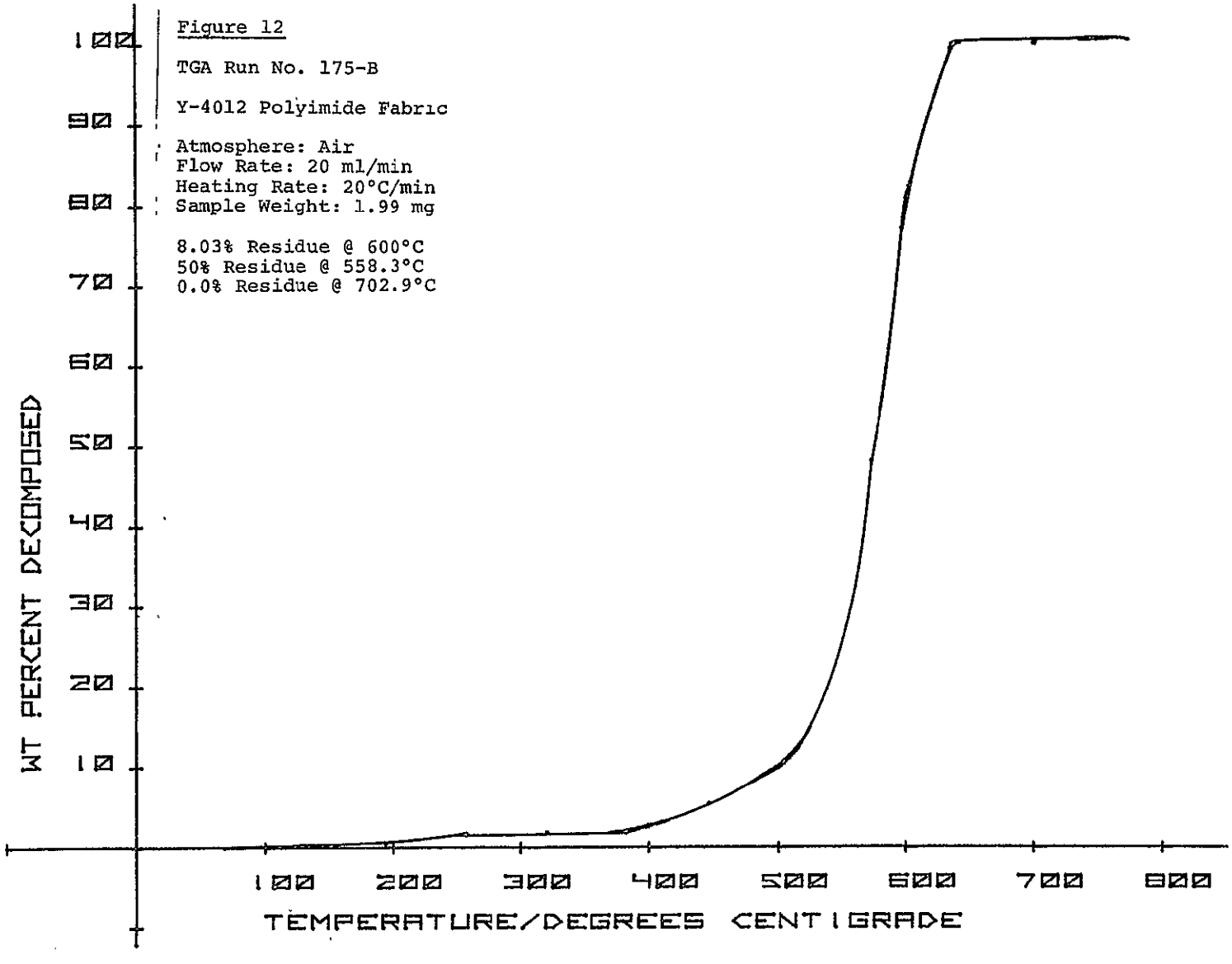


Figure 13

TGA Run No. 175-A

Y-4012 Polyimide Fabric

Atmosphere: Nitrogen
Flow Rate: 200 ml/min
Heating Rate: 10°C
Sample Weight: 10.02 mg

73% Residue @ 600°C
50% Residue @ 663.0°C
10.37% Residue @ 1045.3°C

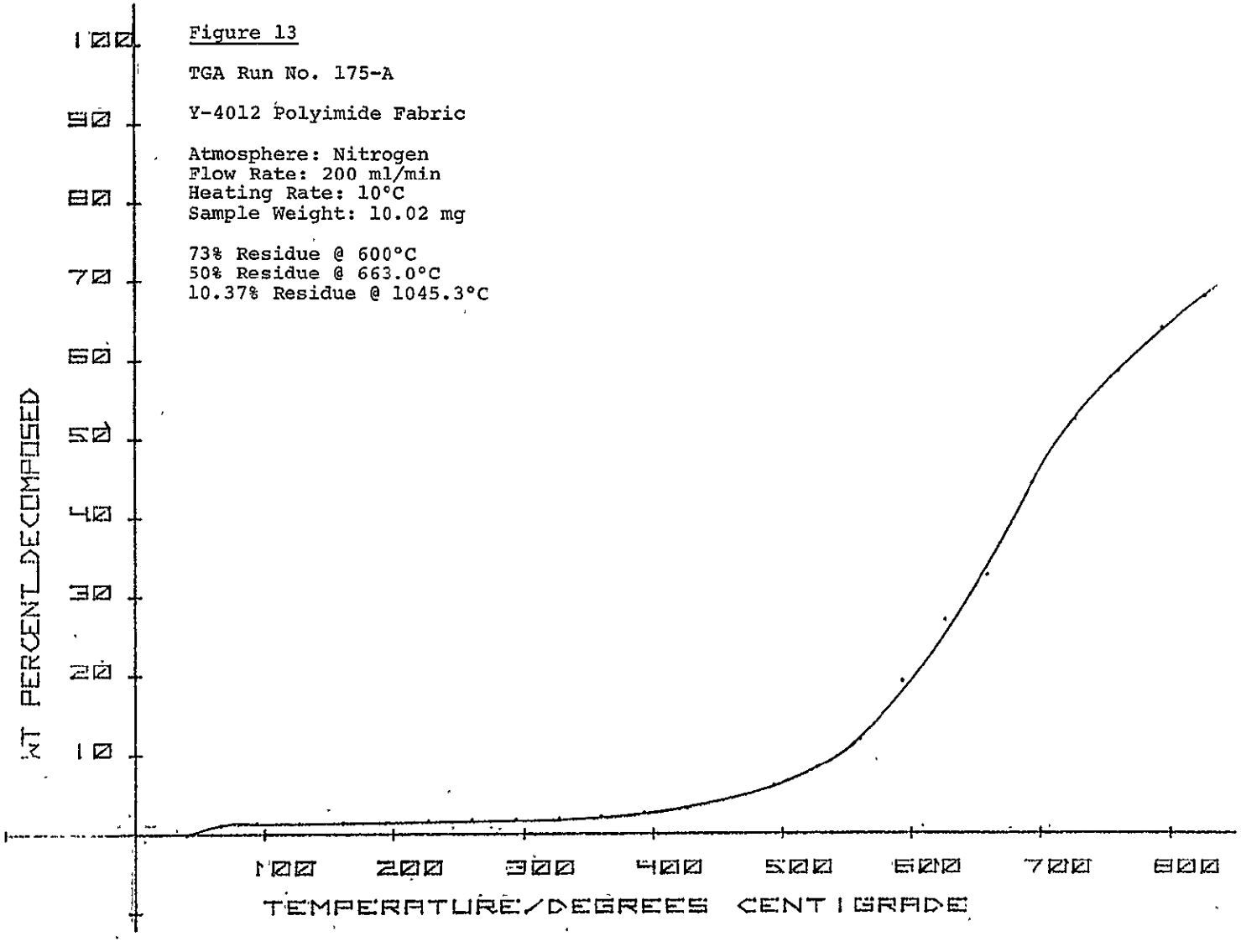


Figure 14

TGA Run No. 177

Y-4012 Polyimide Fabric

Atmosphere: Helium
Flow Rate: 200 ml/min
Heating Rate: 10°C/min
Sample Weight: 9.98 mg

75% Residue @ 600°C
50% Residue @ 860.0°C
10.02% Residue @ 1048.3°C

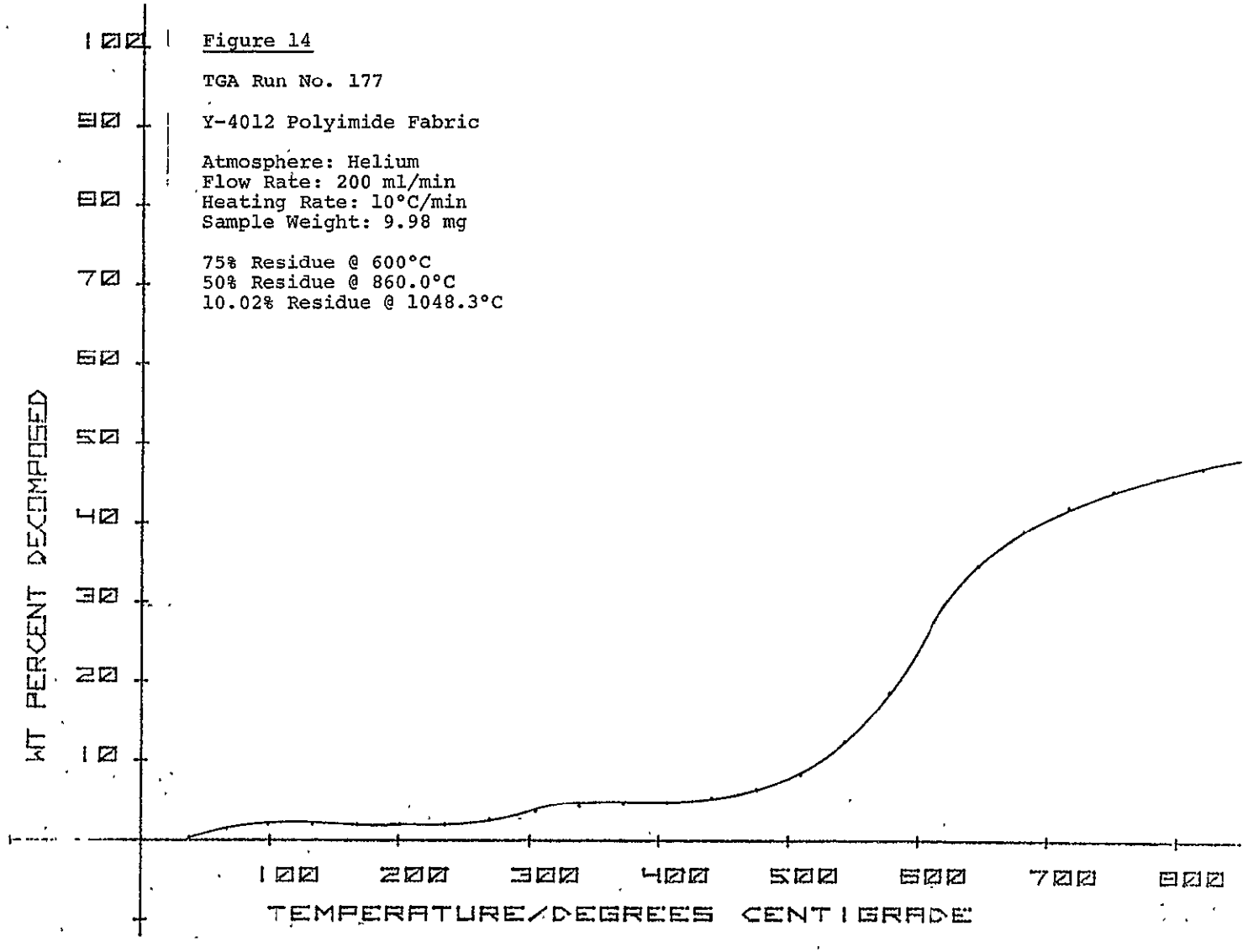


Figure 15

TGA Run No. 178

Y-4012 Polyimide Fabric

Atmosphere: Helium
Flow Rate: 20 ml/min
Heating Rate: 20°C/min
Sample Weight: 1.99 mg

68.5% Residue @ 600°C
50% Residue @ 653.3°C
0.0% Residue @ 922.4°C

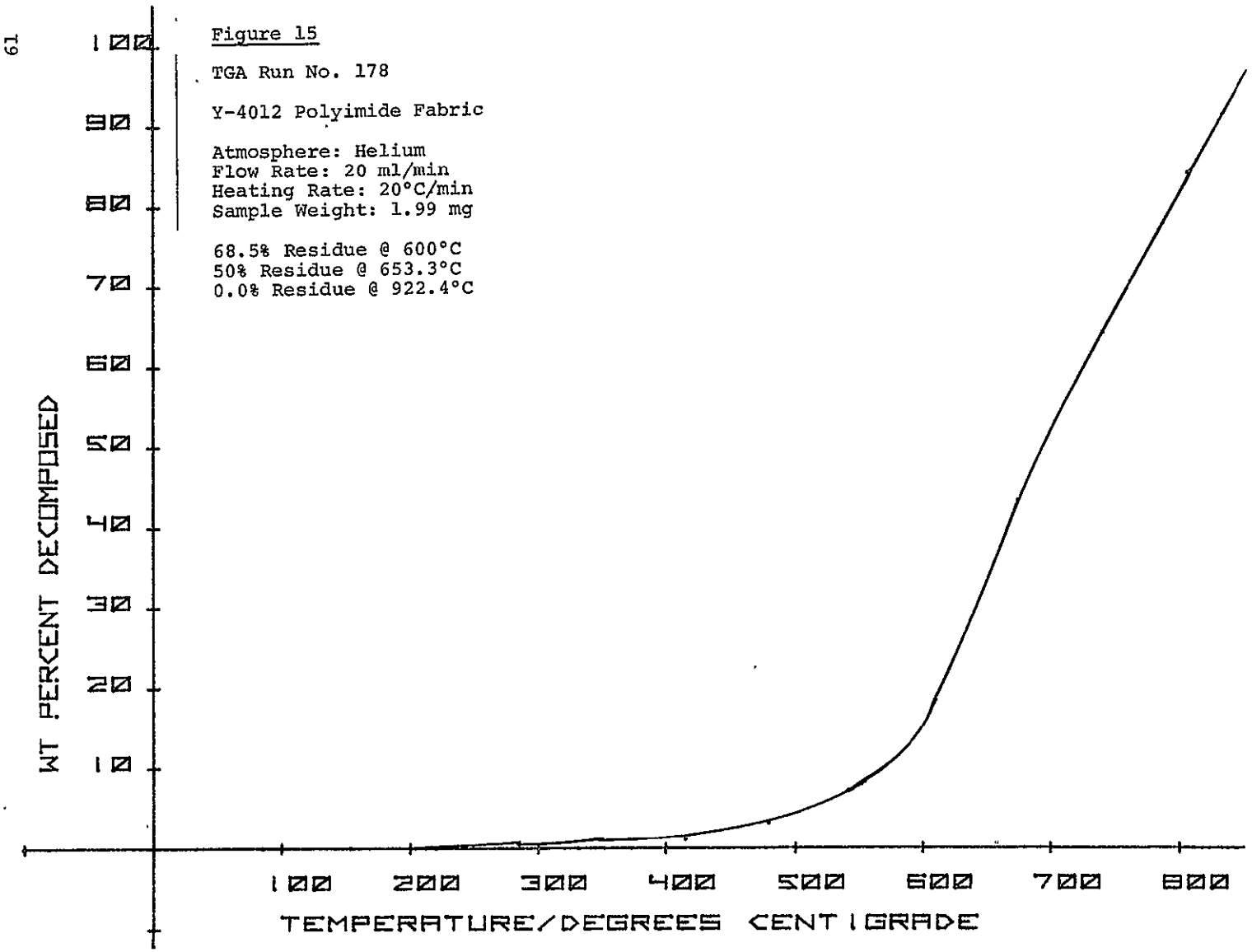


Figure 16

TGA Run No. 182

Y-4120 Tedlar,deglossed

Atmosphere: Air
Flow Rate: 20 ml/min
Heating Rate: 20°C/min
Sample Weight: 2.03 mg

0.0% Residue @ 600°C
50% Residue @ 386.6°C
0.0% Residue @ 838.5°C

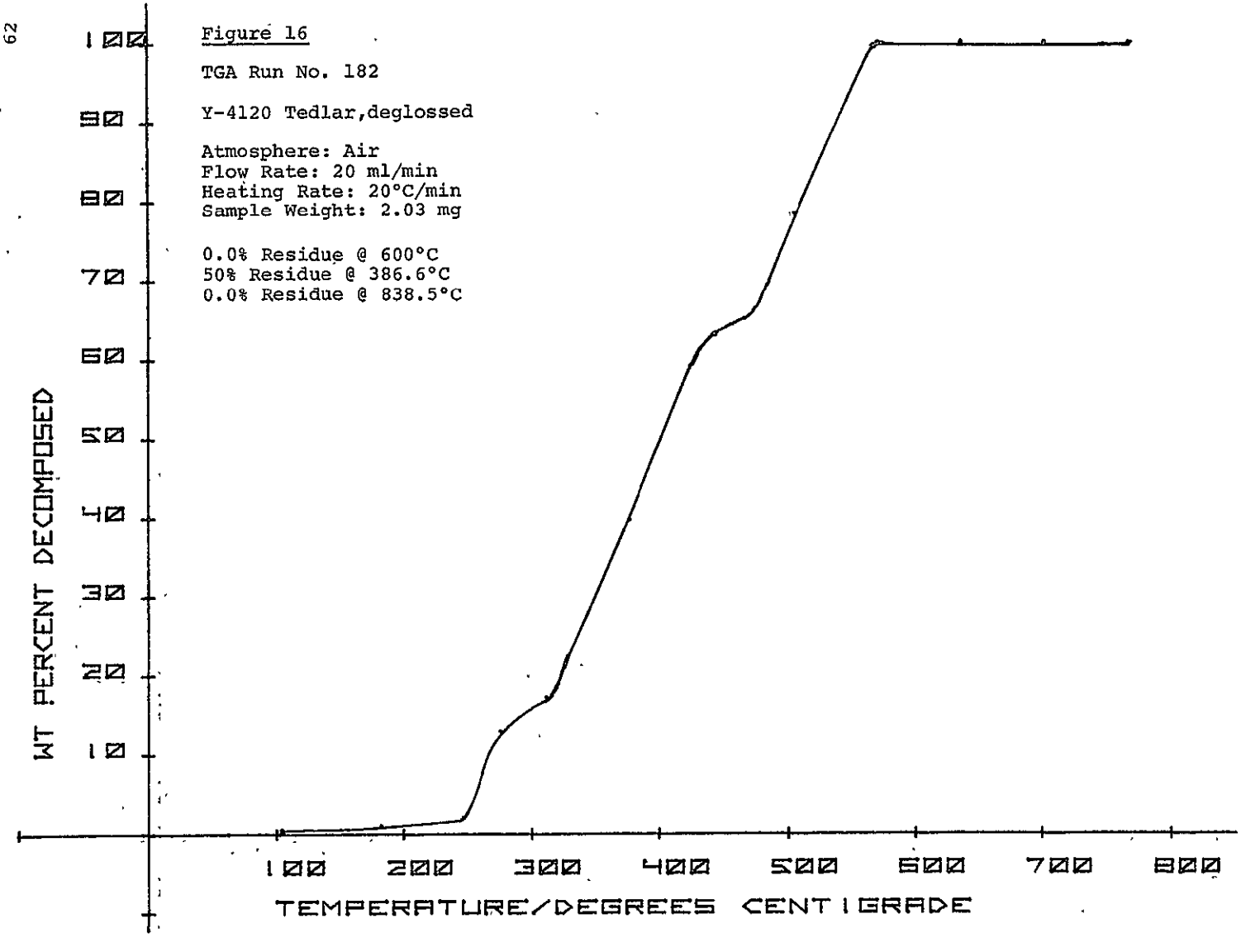


Figure 17

TGA Run No. 303

Y-4120 Tedlar, deglossed

Atmosphere: Air
Flow Rate: 200 ml/min
Heating Rate: 10°C/min
Sample Weight: 2.00 mg

0.0% Residue @ 600°C
50% Residue @ 384.9°C
0.0% Residue @ 811.0°C

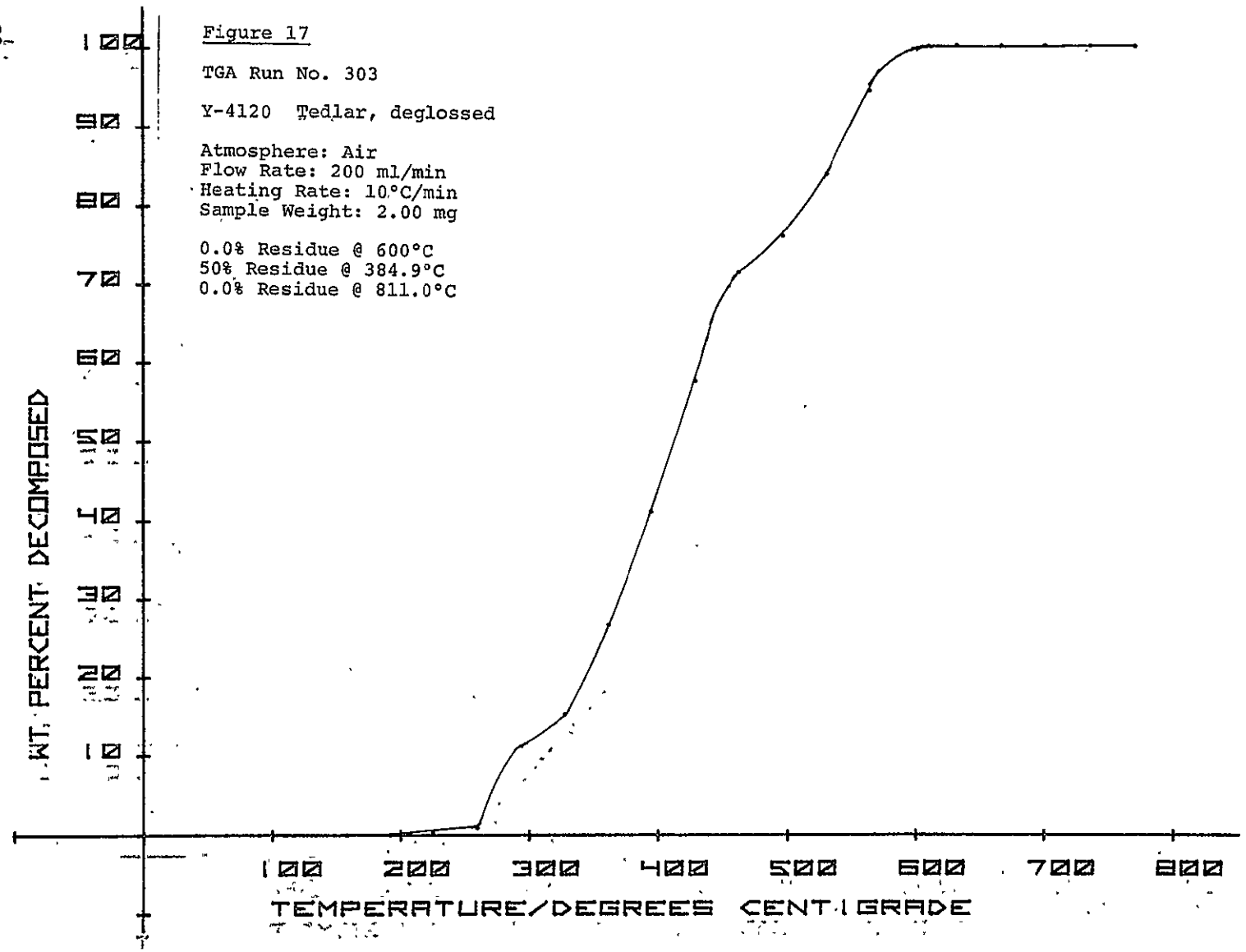


Figure 18

TGA Run No. 189

Y-4157 Kel-F on Nylon

Atmosphere: Air
Flow Rate: 200 ml/min
Heating Rate: 10°C/min
Sample Weight: 2.00 mg

34.5% Residue @ 600°C
50% Residue @ 457.1°C
34.5% Residue @ 1012.6°C

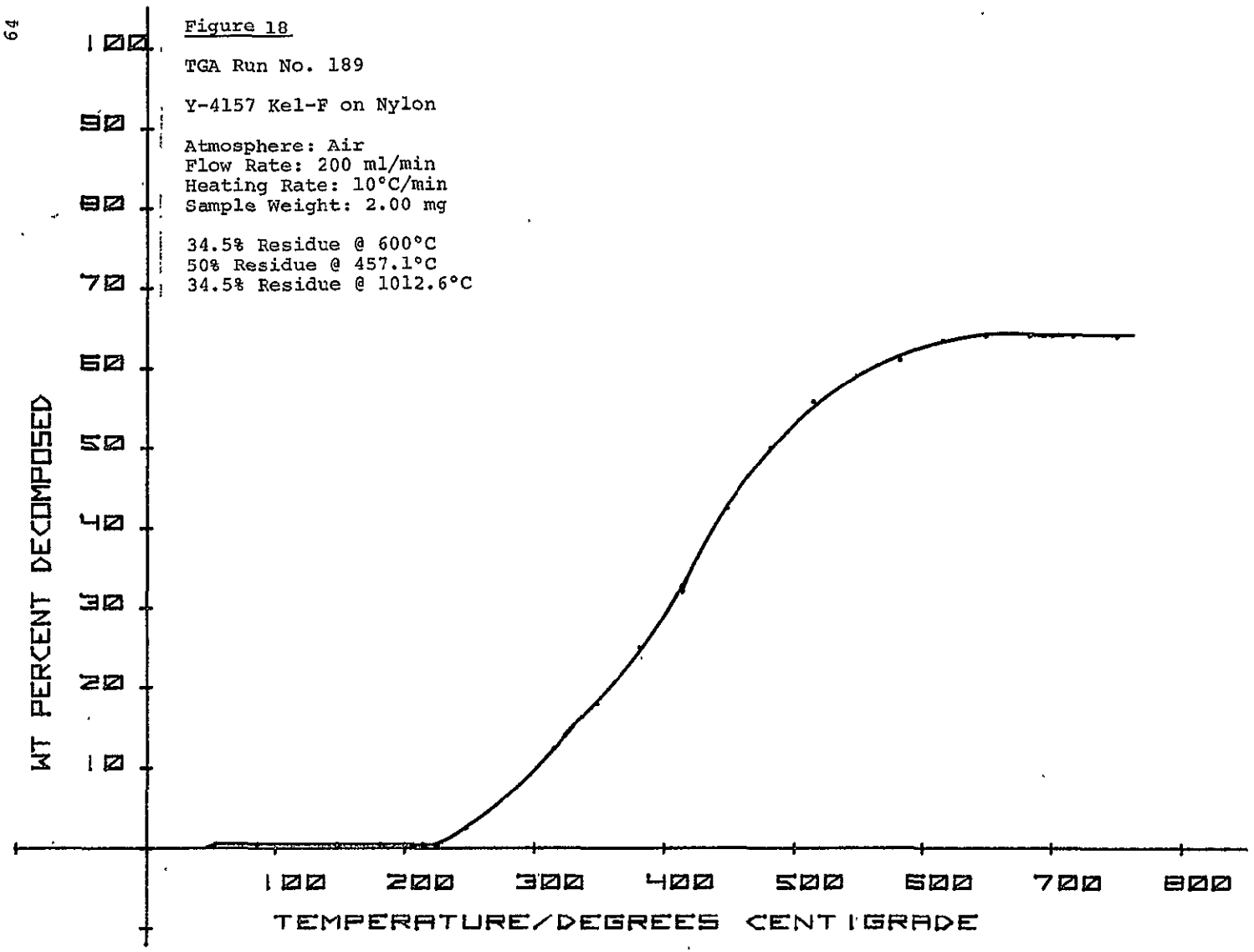


Figure 19

TGA Run No. 309

Y-4157 Kel-F on Nylon

Atmosphere: Nitrogen
Flow Rate: 200 ml/min
Heating Rate: 10°C/min
Sample Weight: 2.02 mg

53.8% Residue @ 600°C
50% Residue @ 752.6°C
43.1% Residue @ 1004.6°C

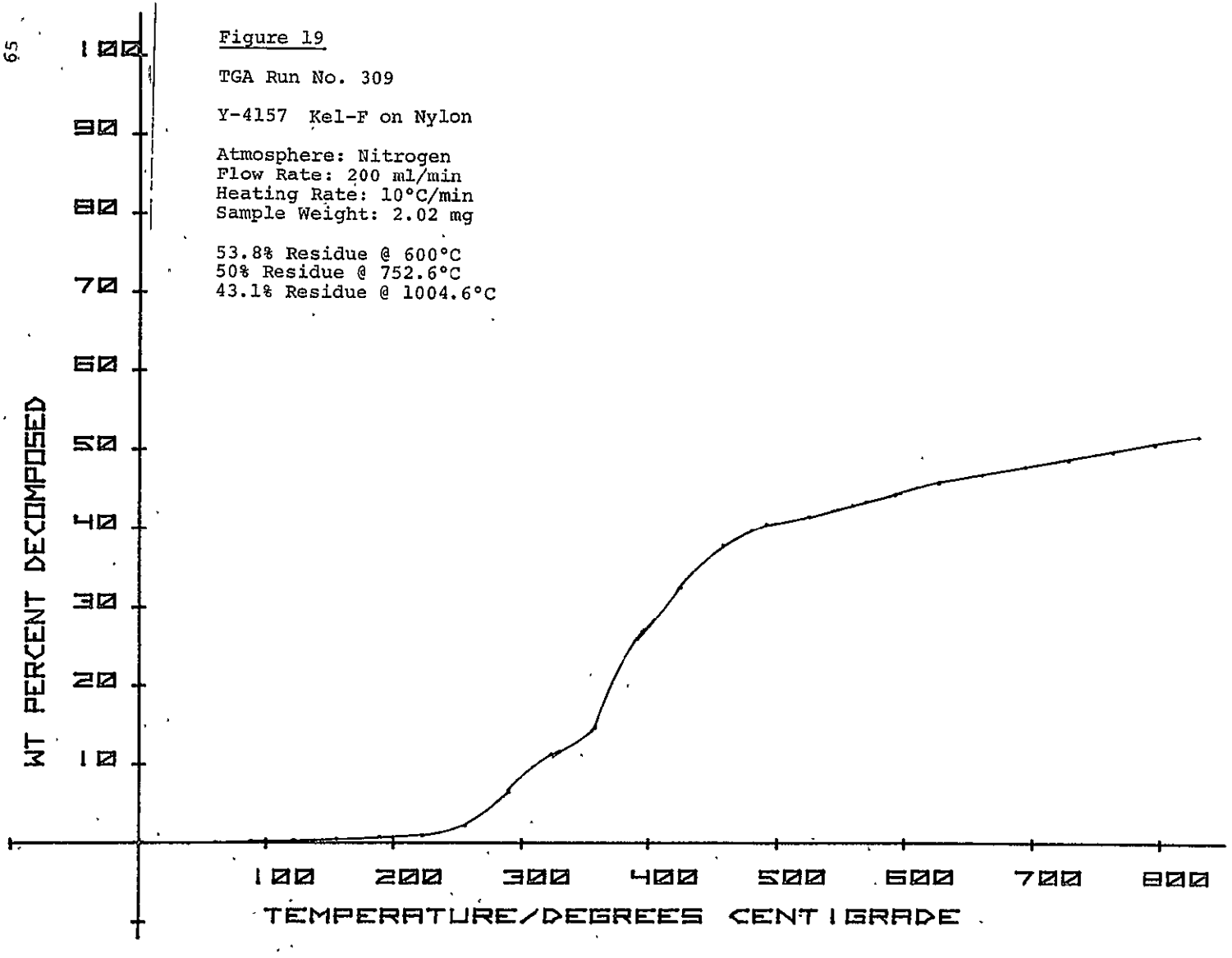


Figure 20

TGA Run No. 217

Y-4157 Kel-F on Nylon

Atmosphere: Helium
Flow Rate: 200 ml/min
Heating Rate: 10°C
Sample Weight: 1.99 mg

53.0% Residue @ 600°C
50% Residue @ 680.6°C
47% Residue @ 767.6°C

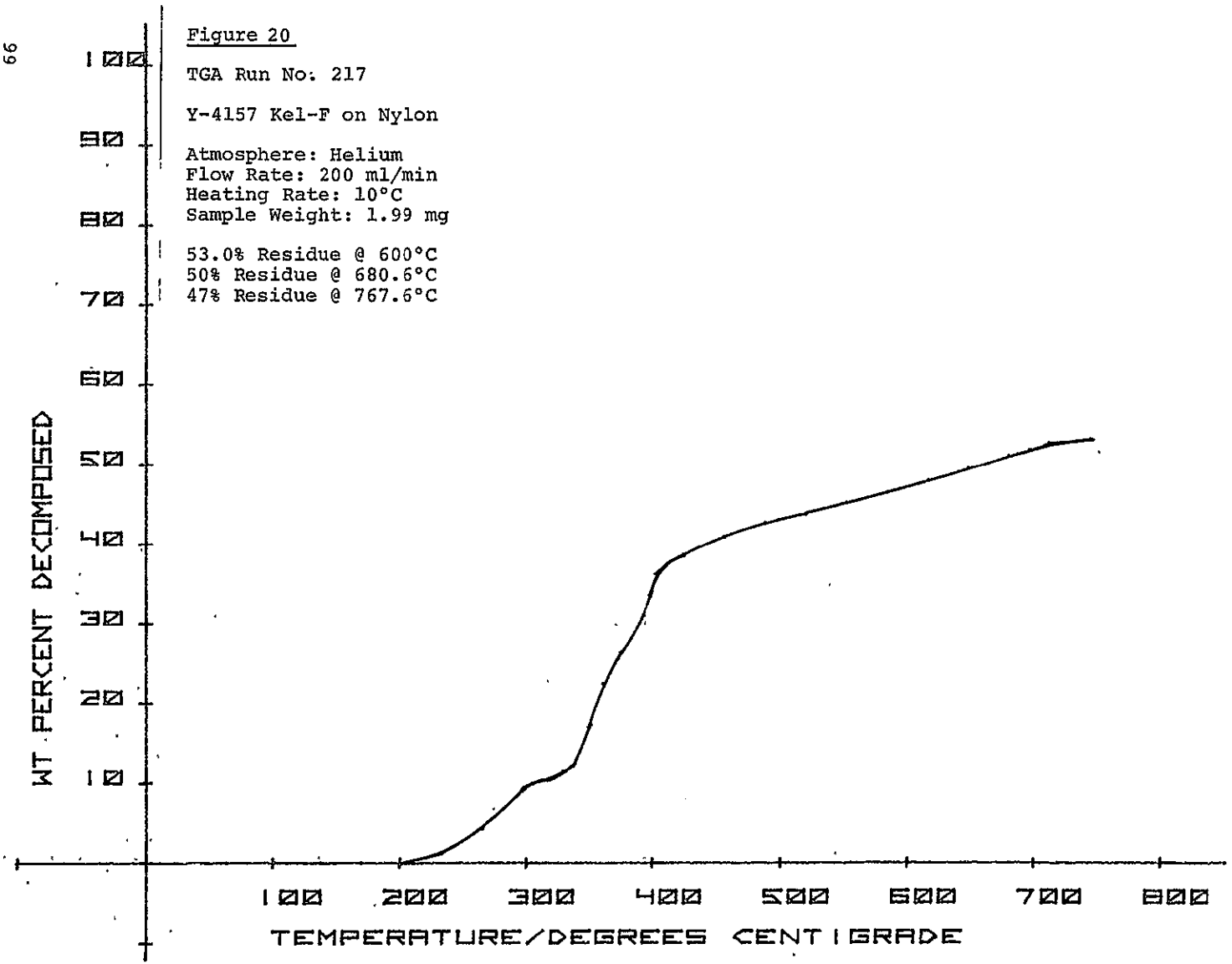


Figure 21

TGA Run No. 186

Y-4158 Nomex Fabric

Atmosphere: Air
Flow Rate: 200 ml/min
Heating Rate: 10°C/min
Sample Weight: 2.05 mg

7.25% Residue @ 600°C
50% Residue @ 495.8°C
7.25% Residue @ 752.6°C

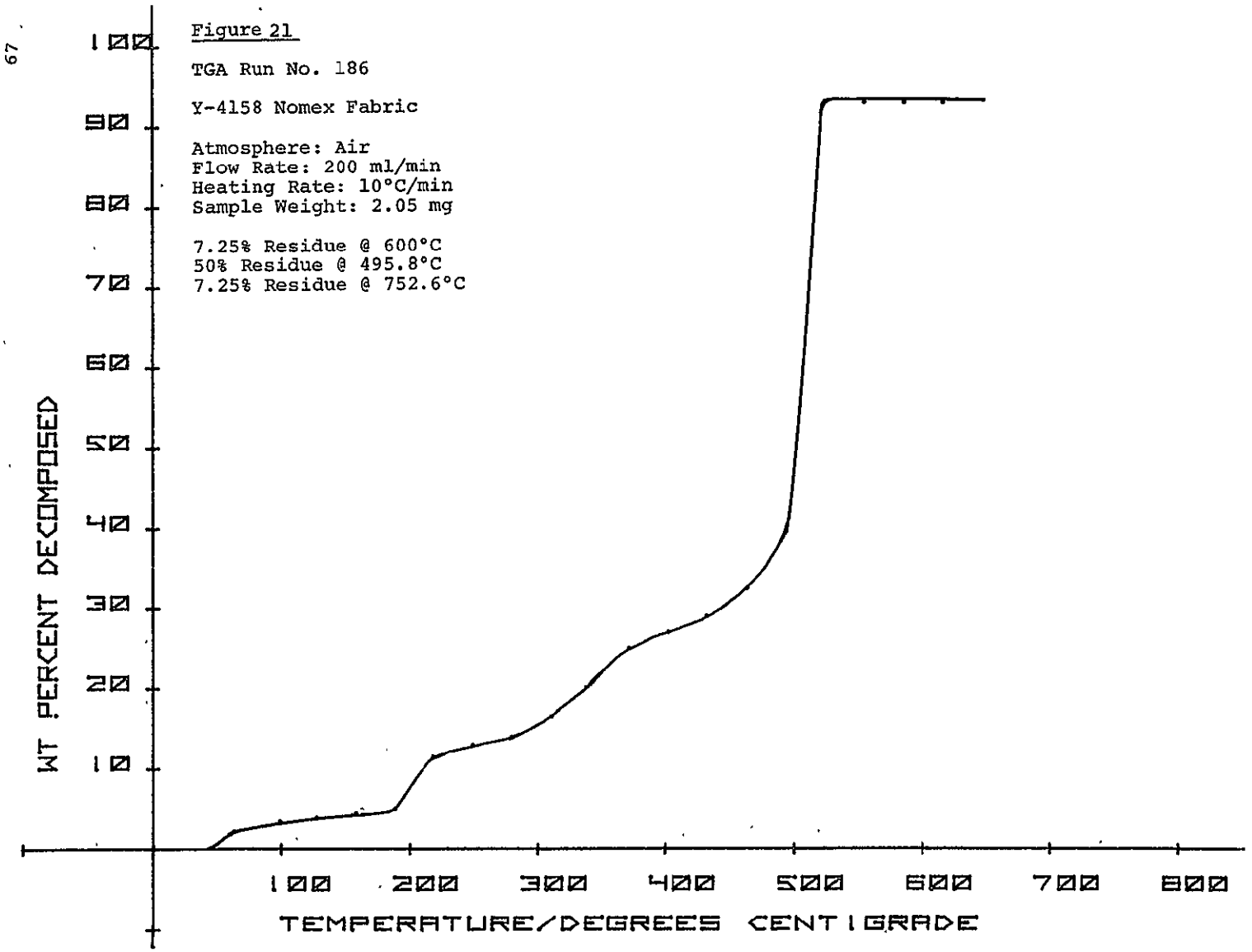


Figure 22

TGA Run No. 311

Y-4158 Nomex Fabric

Atmosphere: Nitrogen
Flow Rate: 200 ml/min
Heating Rate: 10°C/min
Sample Weight: 2.03 mg

51.0% Residue @ 600°C
50% Residue @ 624.3°C
34.5% Residue @ 1021.7°C

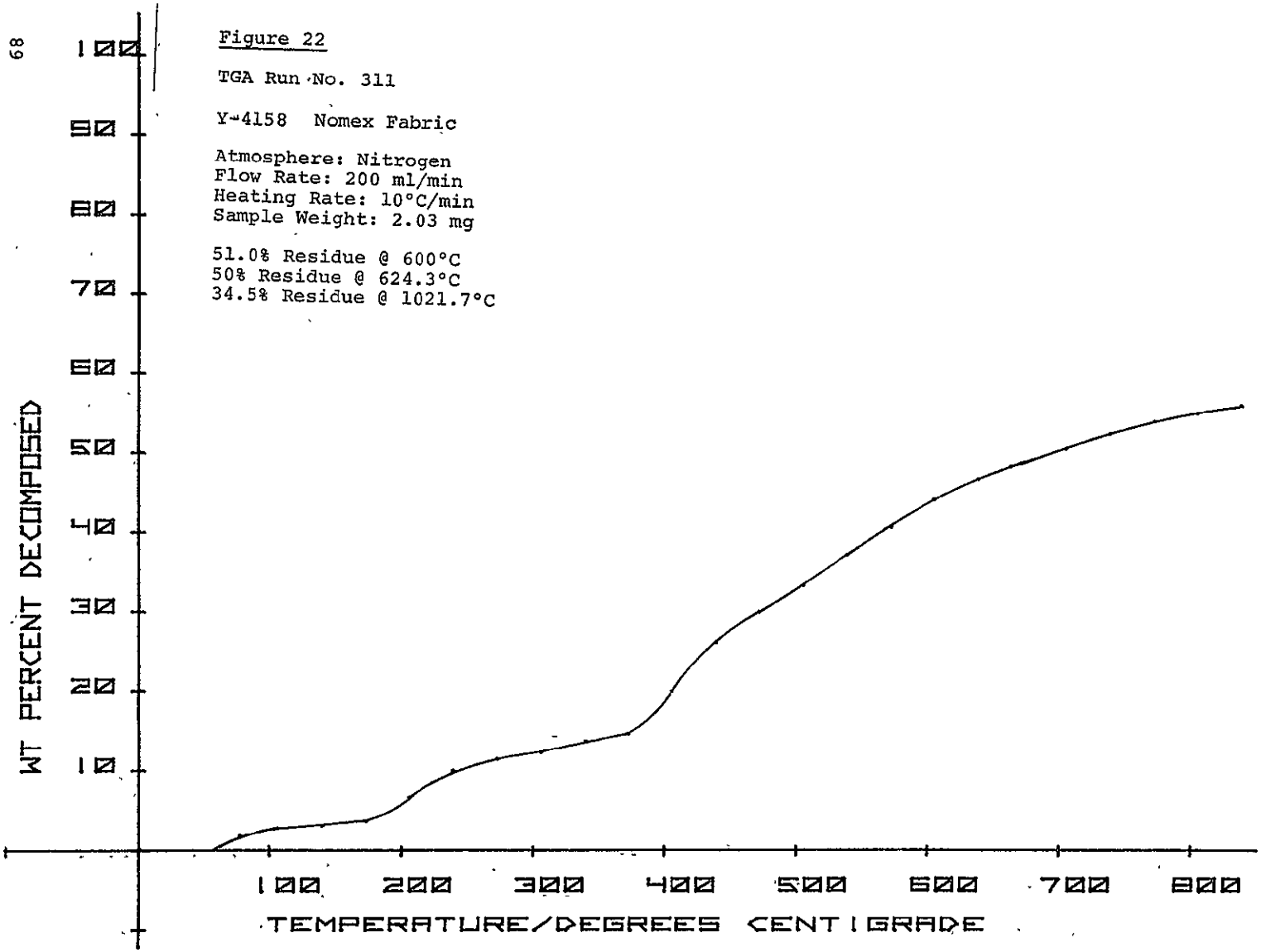


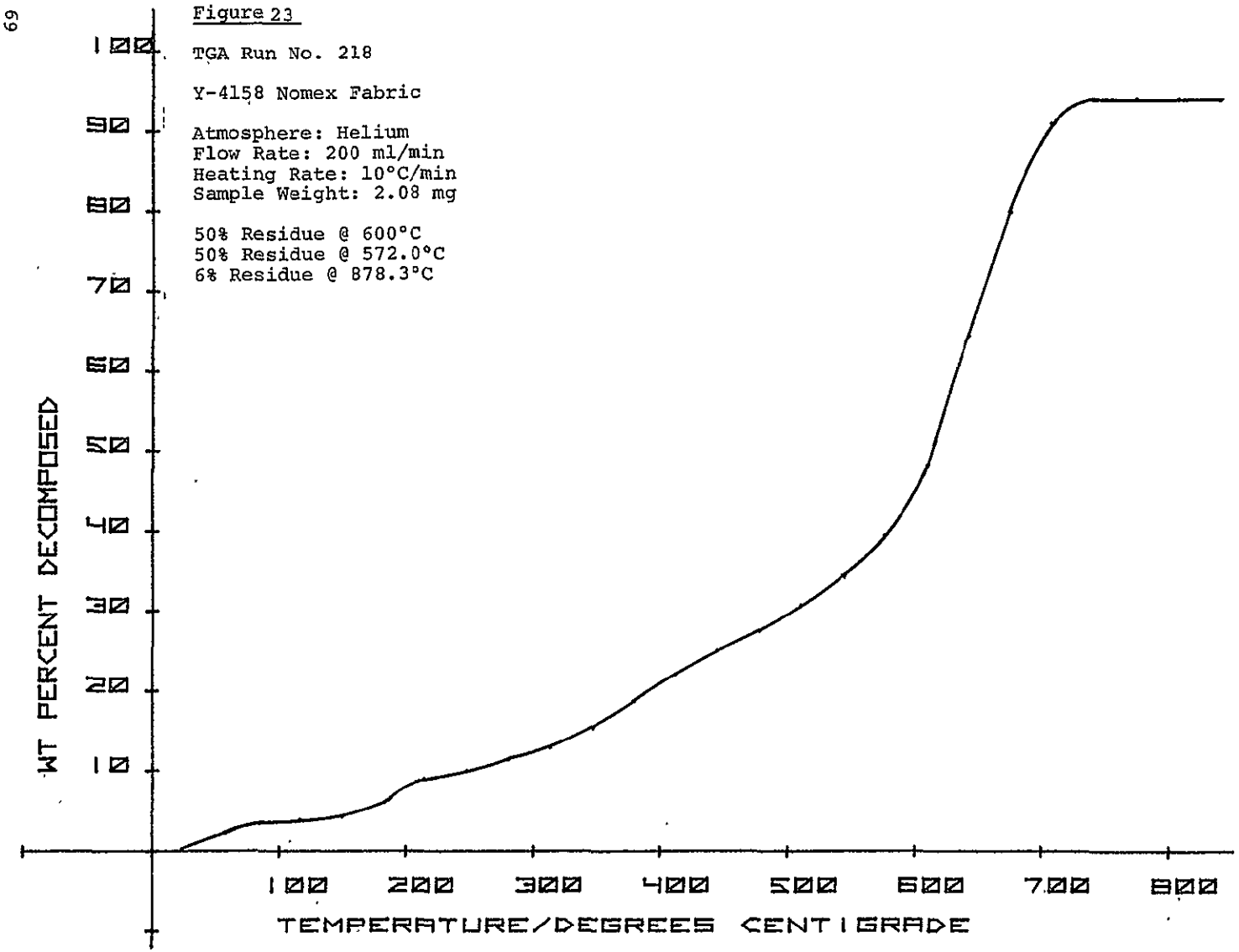
Figure 23

TGA Run No. 218

Y-4158 Nomex Fabric

Atmosphere: Helium
Flow Rate: 200 ml/min
Heating Rate: 10°C/min
Sample Weight: 2.08 mg

50% Residue @ 600°C
50% Residue @ 572.0°C
6% Residue @ 878.3°C



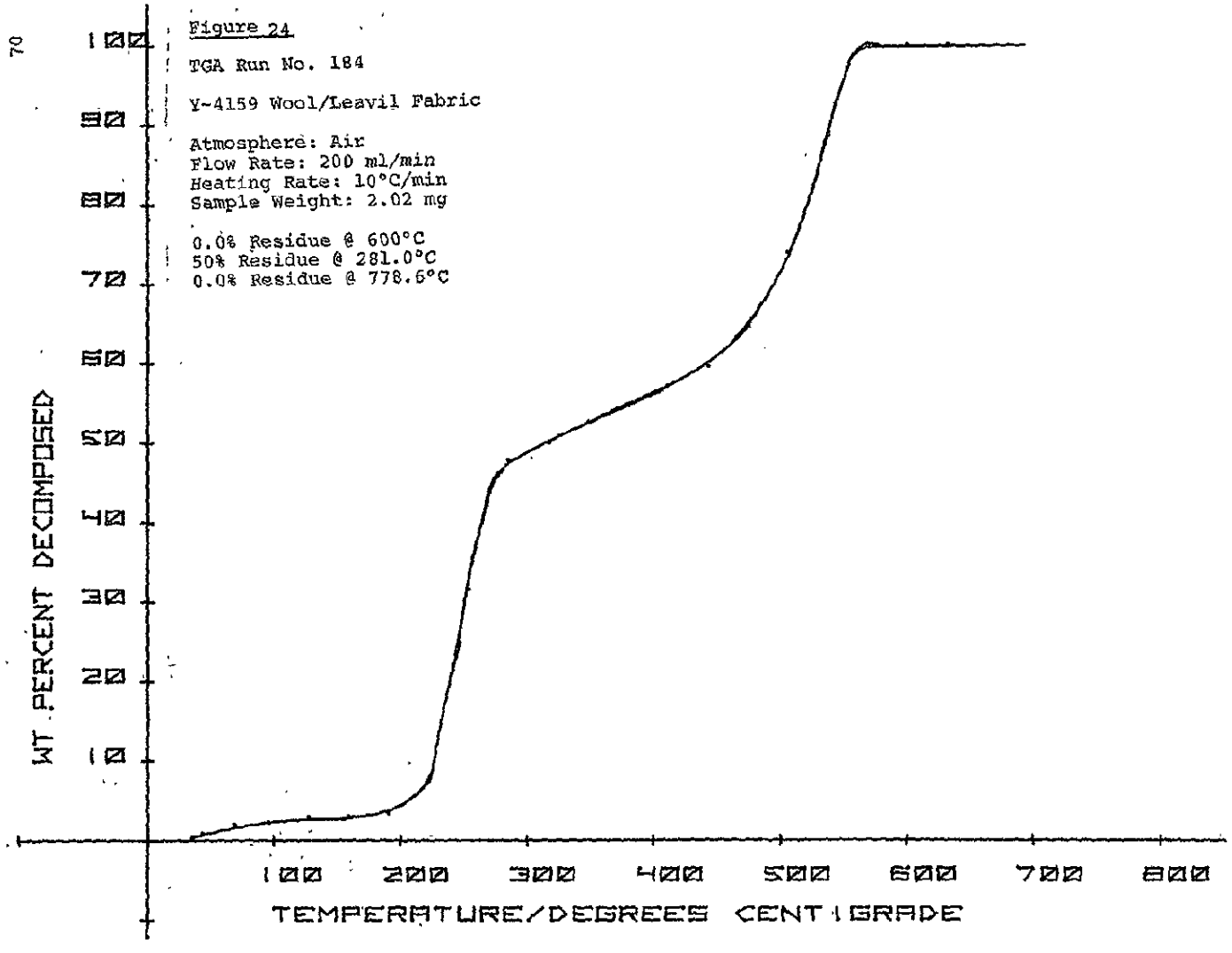


Figure 24
TGA Run No. 184
Y-4159 Wool/Leavil Fabric
Atmosphere: Air
Flow Rate: 200 ml/min
Heating Rate: 10°C/min
Sample Weight: 2.02 mg
0.0% Residue @ 600°C
50% Residue @ 281.0°C
0.0% Residue @ 778.6°C
WT PERCENT DECOMPOSED
100
90
80
70
60
50
40
30
20
10
100
200
300
400
500
600
700
800
TEMPERATURE/DEGREES CENTIGRADE

Figure 25

TGA Run No. 305

Y-4159 Wool/Leavil Fabric

Atmosphere: Nitrogen
Flow Rate: 200 ml/min
Heating Rate: 10°C/min
Sample Weight: 2.12 mg

11.3% Residue @ 600°C
50% Residue @ 317.2°C
5.7% Residue @ 838.3°C

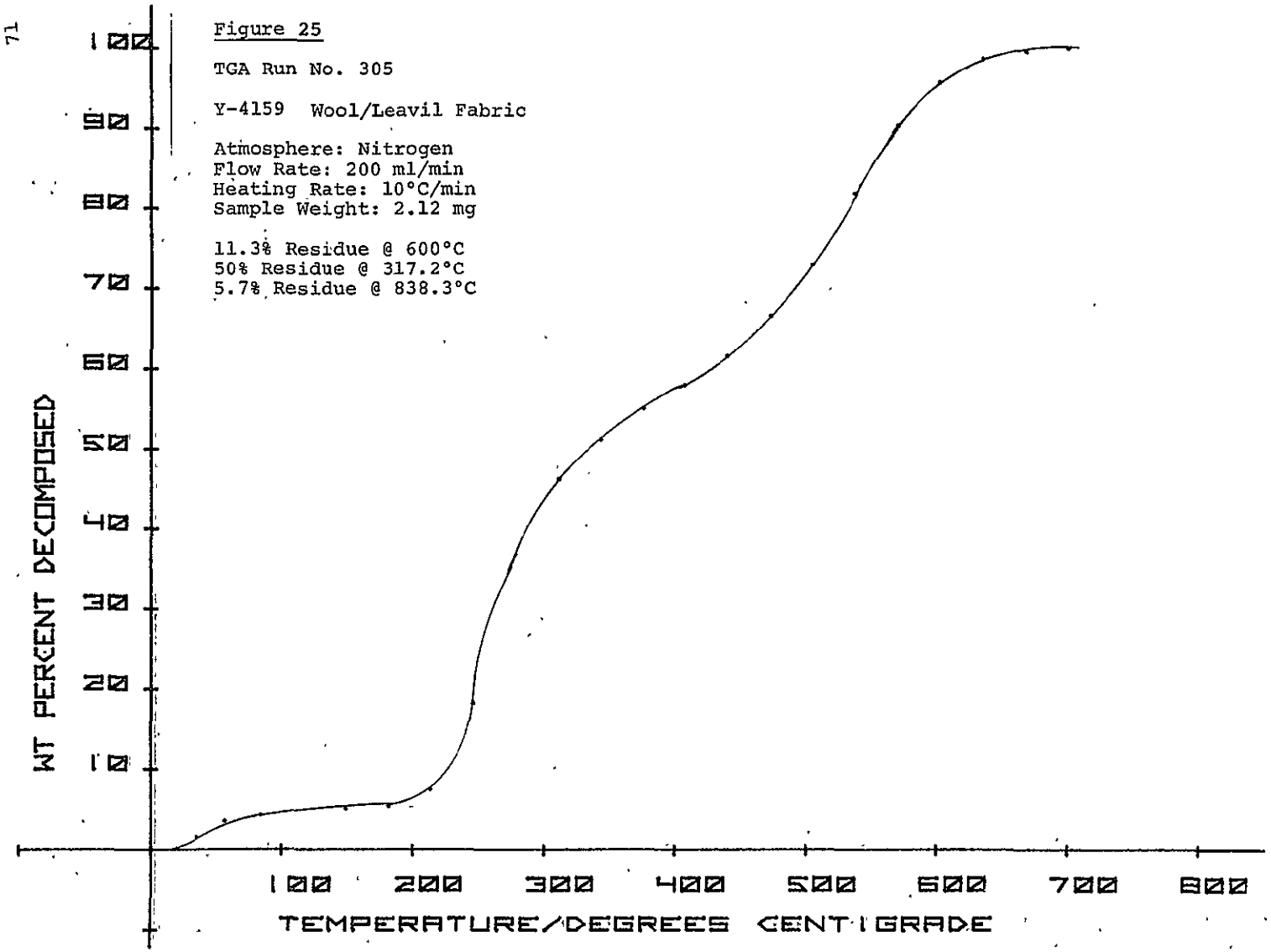


Figure 26

TGA Run No. 221

Y-4397 Decogard x 100

Atmosphere: Air
Flow Rate: 200 ml/min
Heating Rate: 10°C/min
Sample Weight: 2.04 mg

85% Residue @ 600°C
50% Residue @ 561.8°C
82% Residue @ 1001.1°C

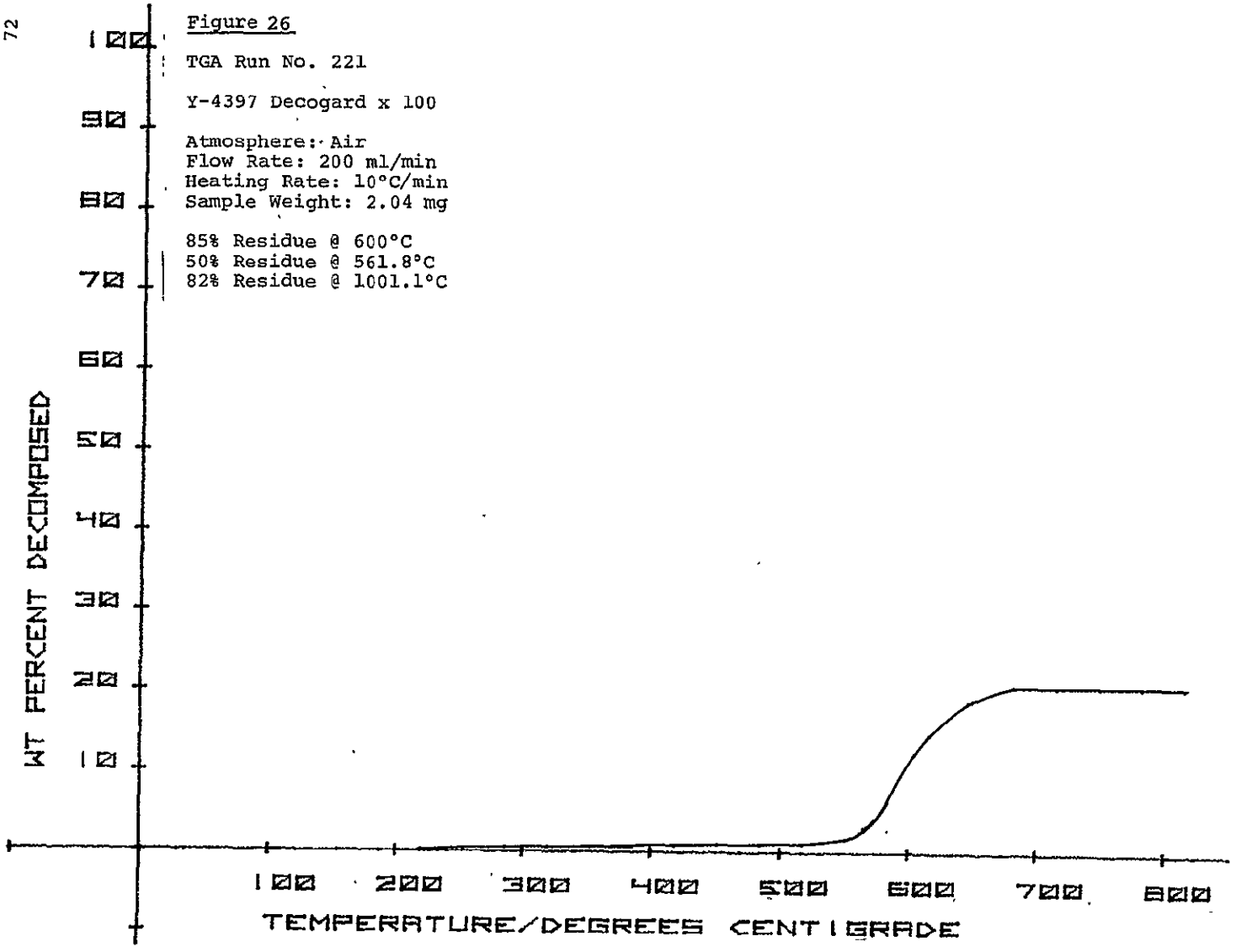


Figure 27

TGA Run No. 301

Y-4397 Decogard x 100

Atmosphere: Air
Flow Rate: 20 ml/min
Heating Rate: 20°C/min
Sample Weight: 2.17 mg

89.9% Residue @ 600°C
50% Residue @ 609.3°C
84.3% Residue @ 945.9°C

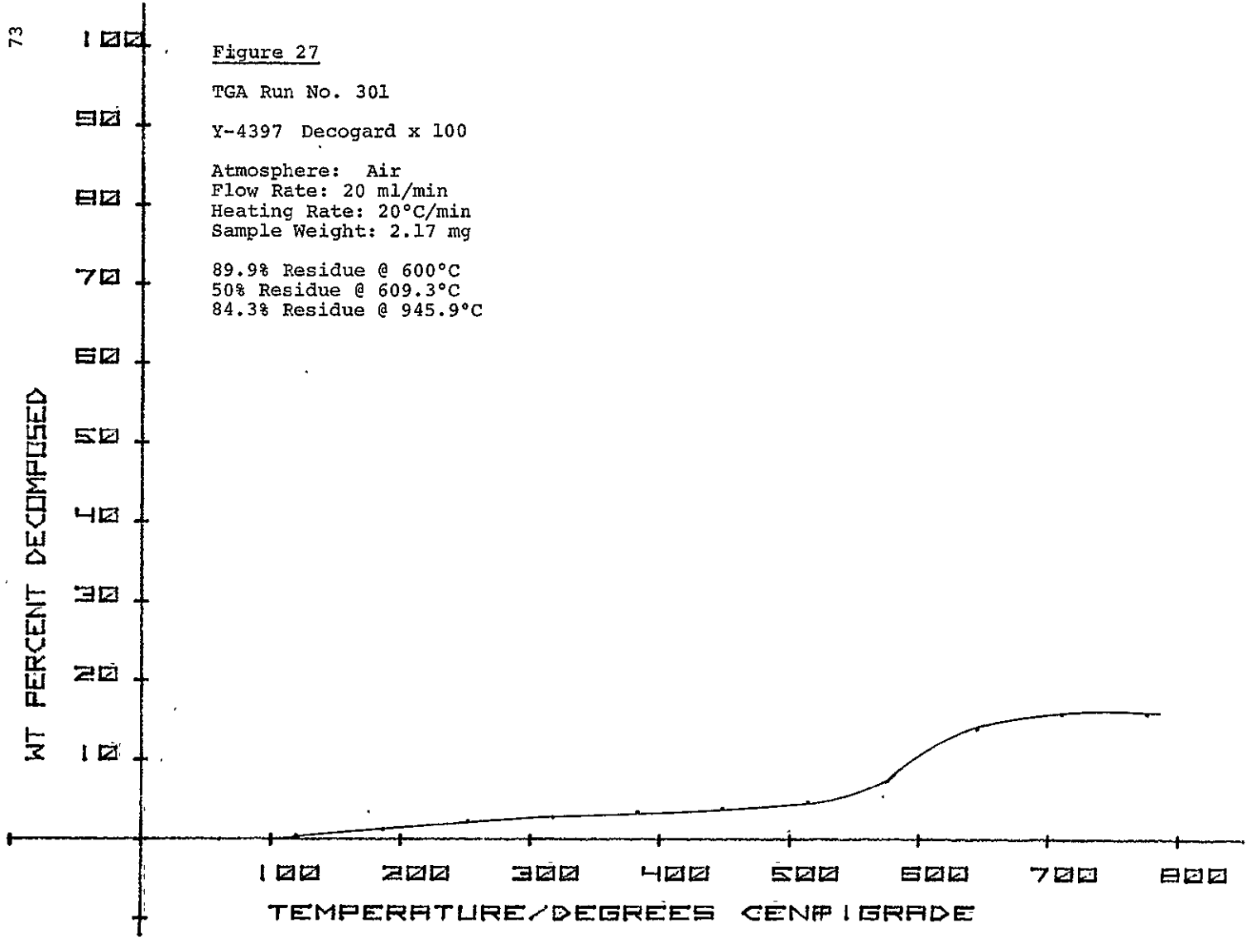


Figure 28

TGA Run No. 307

Y-4397 Decogard x 100

Atmosphere: Nitrogen
Flow Rate: 200 ml/min
Heating Rate: 10°C/min
Sample Weight: 2.07 mg

94.7% Residue @ 600°C
50% Residue @ 633.2°C
85.5% Residue @ 1014.3°C

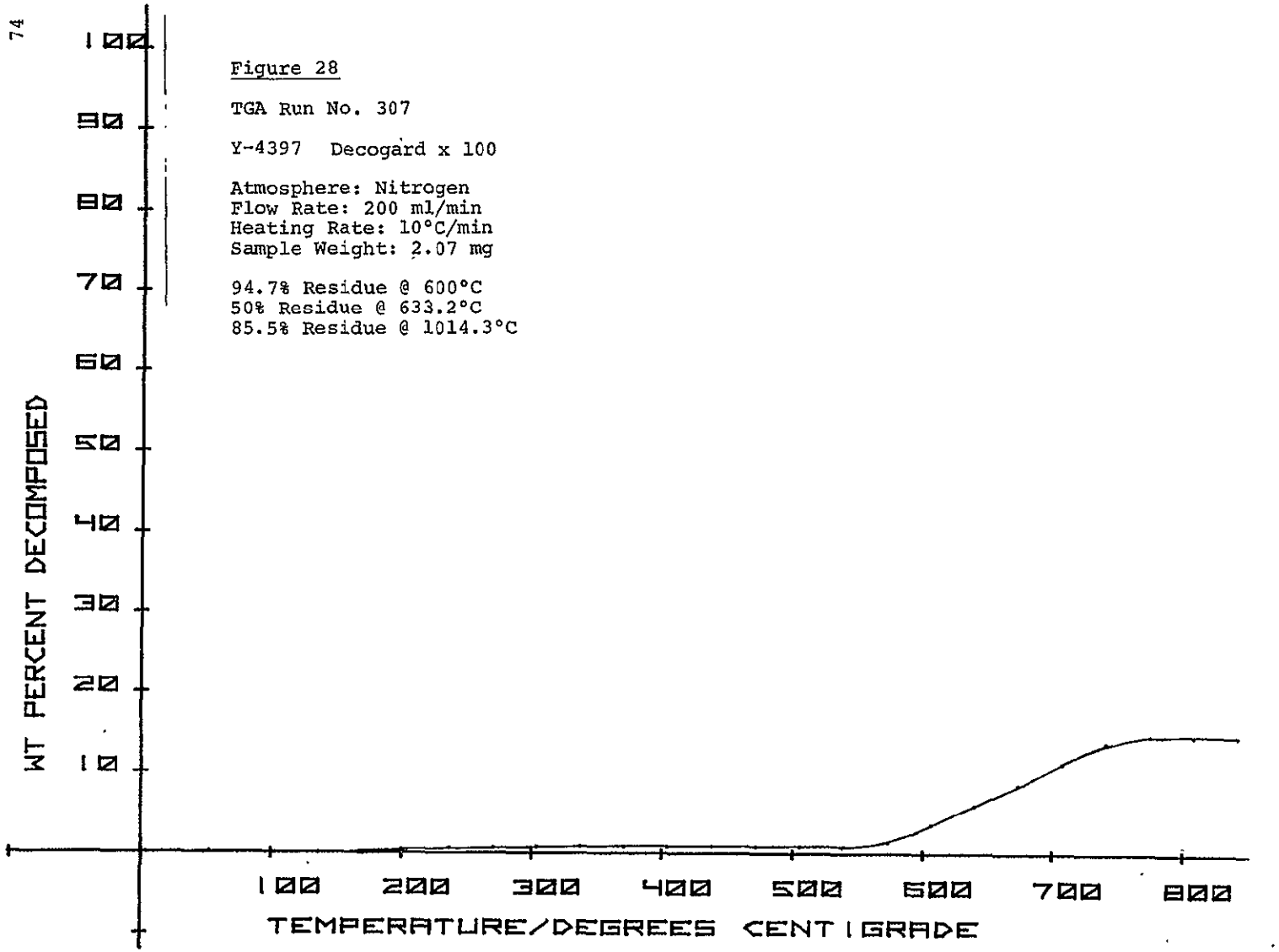


FIGURE 29

MEAN PERCENT CARBOXYHEMOGLOBIN ($COHb$) VS. PERCENT CHAMBER DEATHS

MSTL PROCEDURE

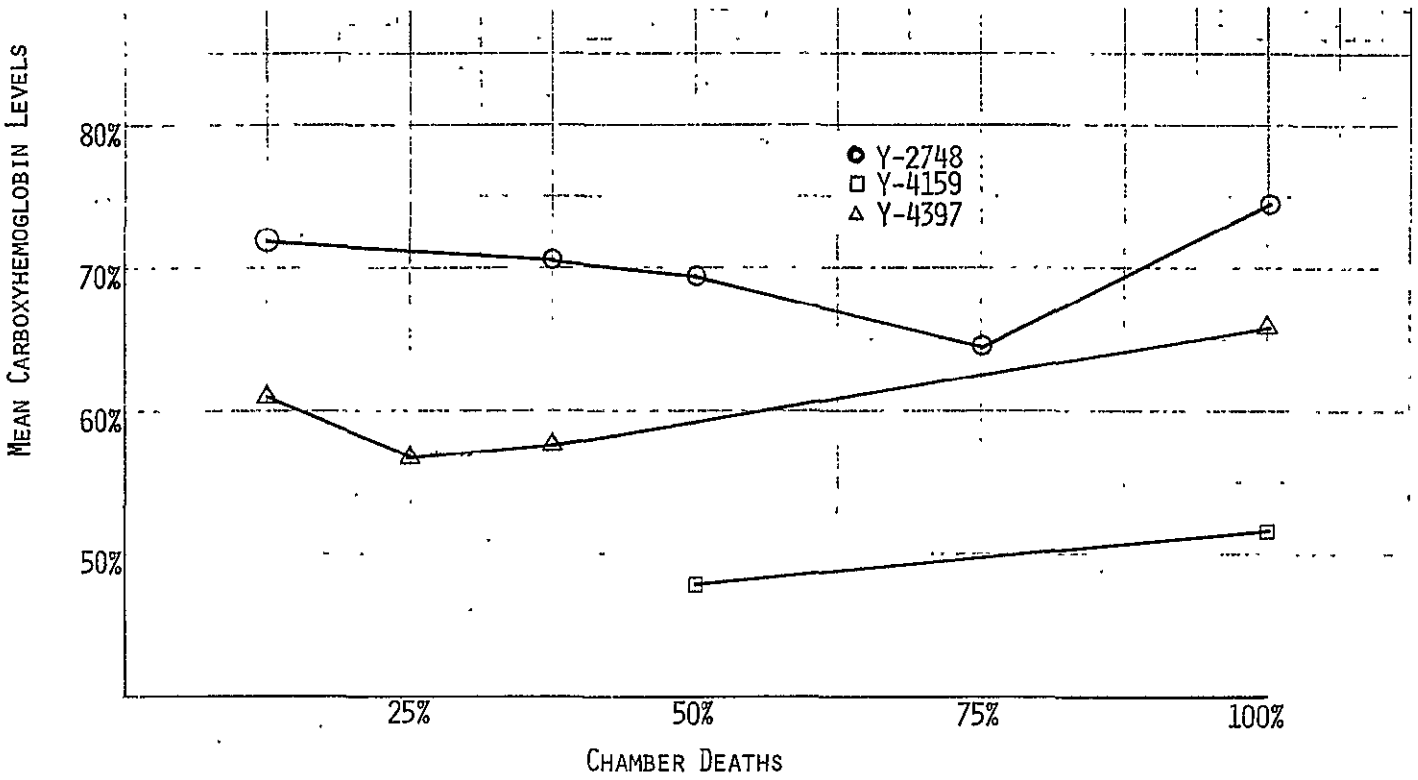


FIGURE 30

MEAN PERCENT CARBOXYHEMOGLOBIN (COHb) VS. PERCENT CHAMBER DEATHS

MSTL PROCEDURE

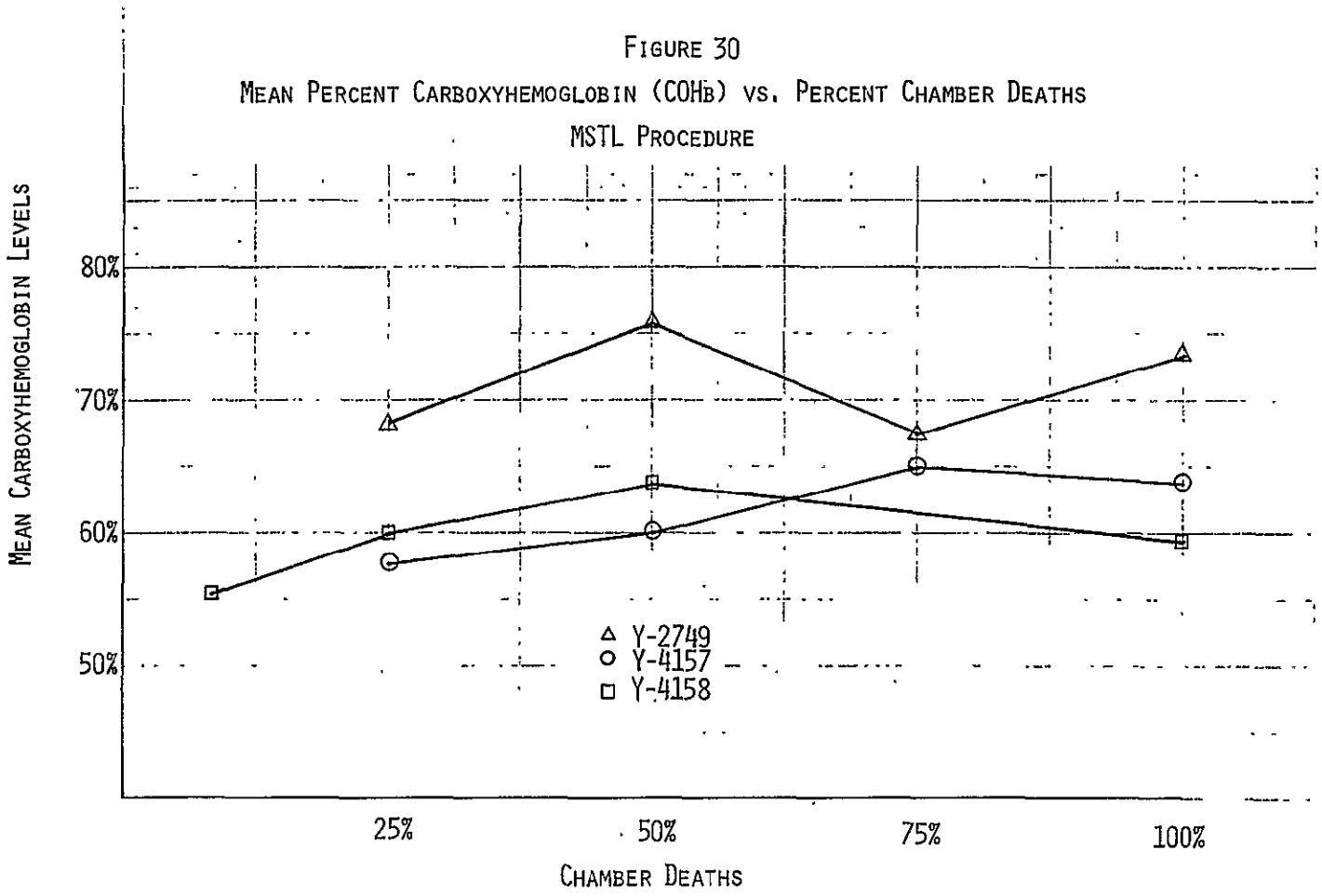


FIGURE 31

MEAN PERCENT CARBOXYHEMOGLOBIN ($COHb$) VS. PERCENT CHAMBER DEATHS

MSTL PROCEDURE

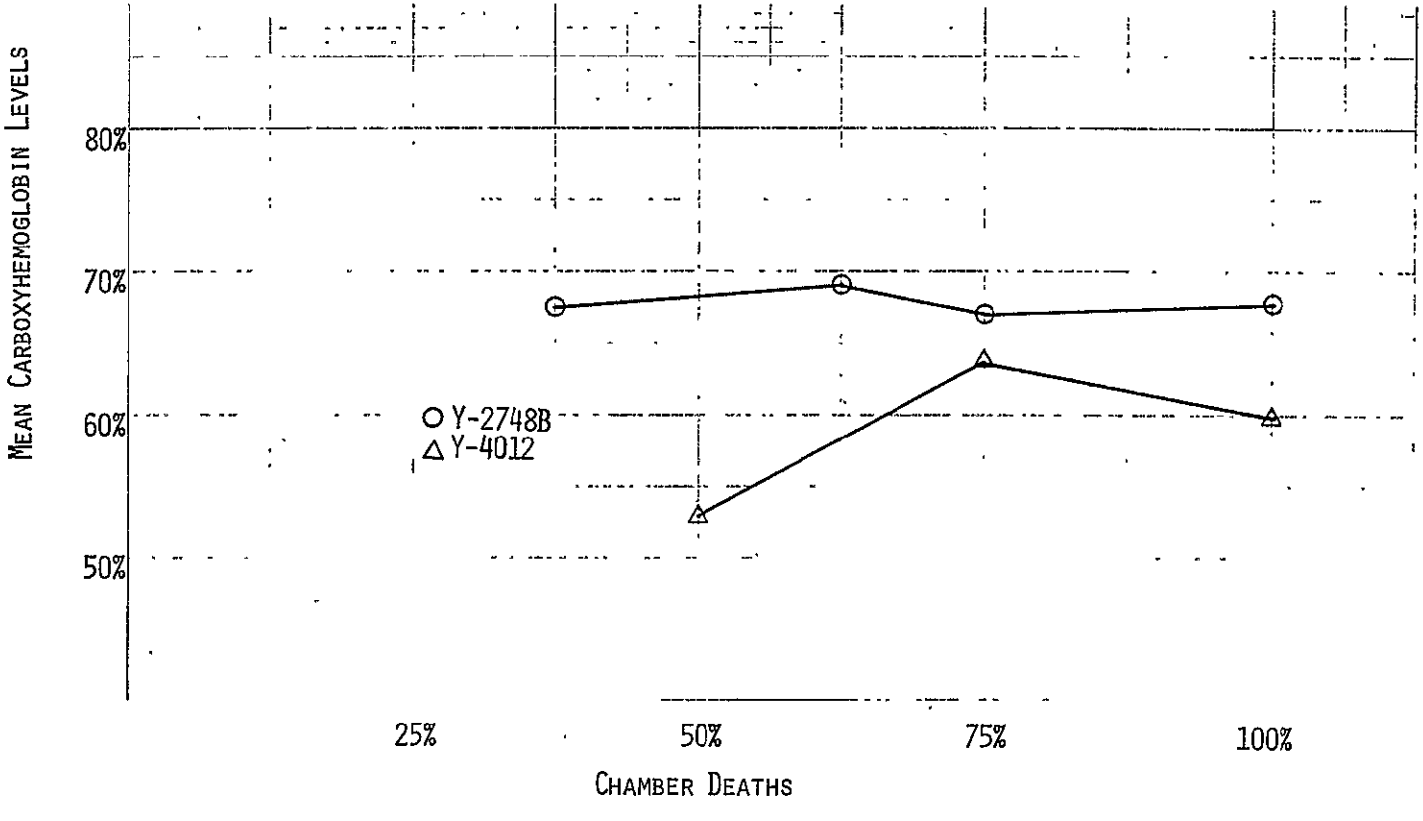
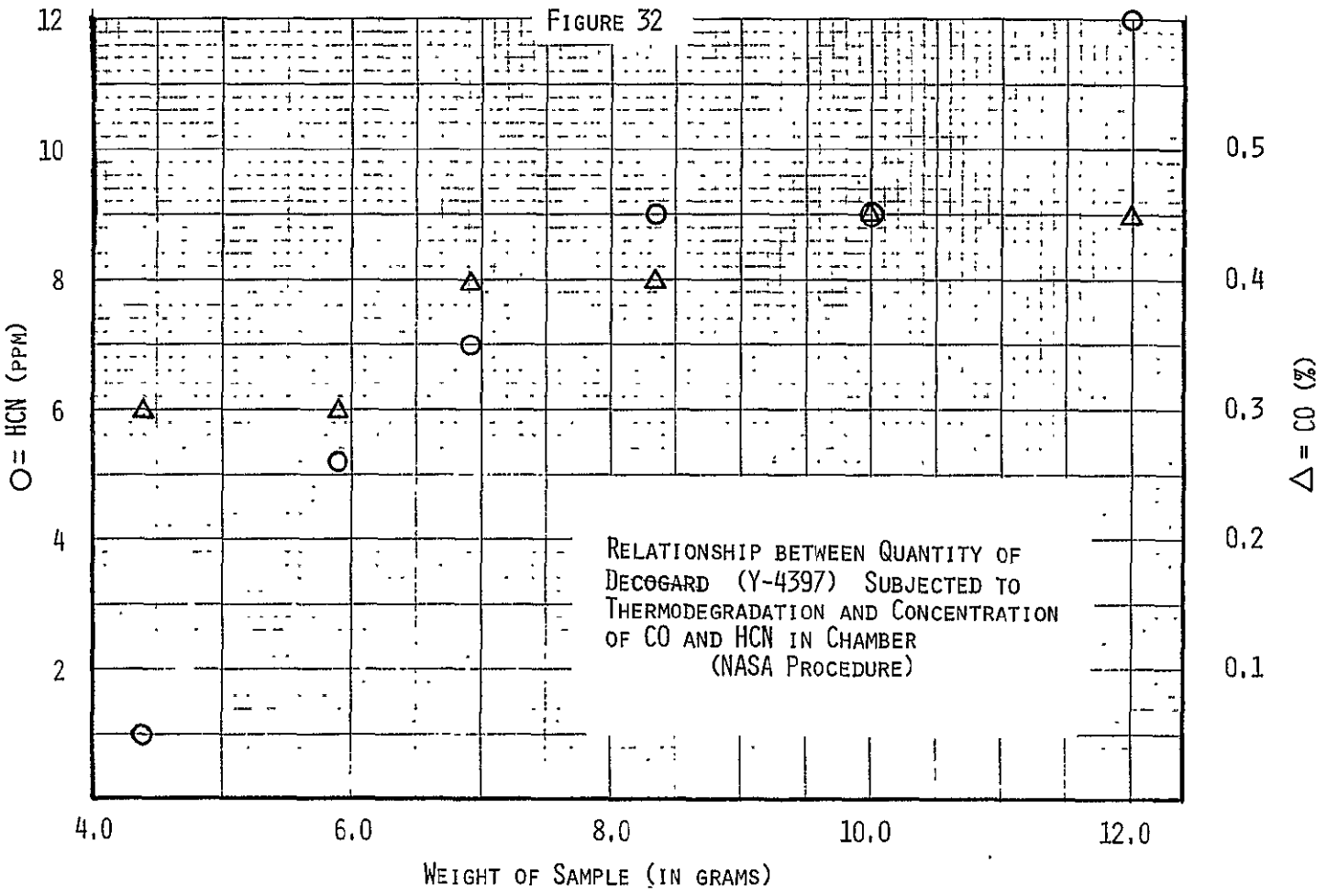


FIGURE 32


12
10
8
6
4
2
○ = HCN (PPM)
0.5
0.4
0.3
0.2
0.1
△ = CO (%)
4.0
6.0
8.0
10.0
12.0
WEIGHT OF SAMPLE (IN GRAMS)
RELATIONSHIP BETWEEN QUANTITY OF DECOGARD (Y-4397) SUBJECTED TO THERMODEGRADATION AND CONCENTRATION OF CO AND HCN IN CHAMBER (NASA PROCEDURE)